

FINAL PROJECT REPORT

(Period: January, 2016 - July, 2019)

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Project Title:

*Development of Appropriate Product by Studying the
Possible Use of Coir Dust in Oil Industries with
Reference to North-East India for Absorption of Oil Spill.*

A Collaborative project from

COIR BOARD

(Ministry of Micro, Small and Medium Enterprises)

Govt. of India

Kalavoor – 688522, Alleppey



उत्तर-पूर्व विज्ञान तथा प्रौद्योगिकी संस्थान

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INDIA

Connecting Science & Technology for a Brighter Tomorrow

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COMPLETION REPORT OF EXTERNALLY FUNDED PROJECTS (2016-2019)

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EXECUTIVE SUMMARY

The present research successfully explored the possibilities of utilizing coir dust, a waste product generating from the coconut husk and fibres, as development of a useful product that could use for clean-up oil pollution i.e. Oil spillage which is the major sources that contribute to environment pollution in oil industries. The Studies undertaken in the project to developed product i.e. Oil Absorbent Particles, Oil Absorbent Mat/sheet, Oil Absorbent block/ bricks/ board of those waste for defend the soil from oil spill and also minimize the coir waste material.

The project works studies the characterisation of coir dust i.e. Proximate analysis, lignin content, cellulose content, pH value, physico-chemical treatments etc. were done to enhance the hydrophobicity and oleophilic character of the coir dust. Coir dust was then exposing to water and crude oil to determine hydrophobicity and oil sorption capacity. After that application in industrial product form, design and fabricate waste coir dust as Oil Absorbent Mat (OAM) which is made from coir dust (CD) in combination with commonly available natural fibre i.e. cotton waste (CW) and jute waste (JW) in the ratio 80:20, 85:15, 90:10. The physical strength of the mats were found to be maximum at 85:15, having tensile strength 43.86 ± 3.5 and 49.17 ± 4.0 MPa for composite of CD and CW, and CD & JW respectively. Similarly, oil absorption also recorded 21.8 ± 2.0 and 19.6 ± 2.0 g/g in crude oil. Various mechanical properties of the product, for instance oil absorption, double fold, bursting strength, ultimate tensile strength (UTS) etc. were determined along with thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). CD, CW and JW were characterized by FTIR, XRD, TGA and SEM analysis. Results clearly portray that natural fibre significantly improves the oil absorption, mechanical and thermal properties, and also increases the flexibility of the final composite mat. These mats are an advantage over the commercial ones in terms of higher oil absorbency and physical strength.

Chapter 1

Introduction

Review of Literature:

Oil spill is one of the major sources that contribute to environmental pollution in oil industry. It happens as a consequence of activities related to crude oil exploration, industries, and transportation. On the land, oil spill penetrates to soil matrix and thereby contaminate ground water [1]. The entire process of oil spill pollution is handled worldwide by two ways, restricting oil spill from spreading to the surroundings and removing the spill from contaminated surroundings. Although, petrochemical plants and oil refineries are helpful to society, but they produce a large amount of hazardous waste [2]. Additionally, oil spills during exploration, transportation, and refining, have caused serious environmental problems [3]. Regular physico-chemical methods can quickly remove the majority of spilled oil, but some simply transfers contaminants from one environmental medium to another medium and produce toxic by-products. Moreover, crude oil cannot be completely cleaned up with physicochemical methods [3].

A huge quantity of coir dust i.e. a major by-product is produced, in the processing of coir fibre from the coconut husk and development of product from the processing fibre [4]. Annually around 7.5 million tons coir dust/pith is produced [5]. Increasing of coir waste in the surrounding area of coir industries day by day, has created two vital problems (i) occupied valuable ground space, and (ii) contamination of potable groundwater due to percolation of remaining phenolics from these dumps [5]. It decomposes very slowly in soil as its pentosan-lignin ratio is below 0.5 [6], and due to chemical and structural complexity of lignin cellulose complex [7]. CD is mentioned as that brownish colour with light weight spongy particle which falls out at the time of fibre processing [8]. About 70% of the weight of the coconut husk is coir dust [9]. This coir dust has been disposal by the

burning or dumped without control, otherwise use at crop plant as raw organic manure [10-12]. This burning also creates various environmental problems. It contains higher amounts of cellulose in addition potash and lignin and ability to excellent moisture persevering capacity but decomposition is time-consuming [13]. It's have no any commercial value except, may be, in applications where sawdust is used in a very limited amount. Coir dust, a good oil absorbent media, shows superior quality result the oil absorbency [14]. Moreover, Coir pith or dust provides perfect oil absorption medium when, used in small particulate sizes [14]. Coir pith or dust is used to clean up oil spills in solid surfaces, highways, concrete slabs, soils, as well as the surfaces of water bodies [14]. It is reported to be composed of cellulose, pentosan, furfural, and lignin with excellent moisture retaining capacity, but is slow in decomposition [13, 15, 16]. However, it is necessary to find an instant solution to the constant problem of coir dust disposal by proper utilization of this waste material.

Since a major portion of this waste is of biological origin, hence the use of these materials in making fruitful products would definitely be supportive from the point of ecosystem restoration. These coir waste materials alone or in combination with natural fibres may produce useful products through certain mechano-chemical processing. Due to the nature of easy availability, cheap, high oil absorption capacity, lower density, minimum energy utilization, biodegradability and renewability of natural fibres, several generations are using it in synthetic products [17]. Generally, the products manufactured from biological source are considered to be environmentally benign [18]. They show various beneficial properties like superior oil absorbing property, physical strength, lower density, which yield light weight absorbent mats that find use in many industrial applications. Natural fibres are also utilized in thermoplastic polymers reinforcement for automotive applications as well as construction materials etc [19-21]. Thus incorporating bio-

renewable materials make the finished product higher oil absorbent, biodegradable, renewable and more eco-friendly.

For effectual use of such industrial waste, a study was undertaken to develop high oil absorbing mat/pad from coir dust in combination with plant fibres like cotton and jute wastes, implementing a simple process using commercially available equipments. The cotton fibre is an ideal oil-absorbing material due to its low density and sloppy internal structure as well as great fluid adsorption space [22]. The physico-mechanical strength properties like UTS, folding endurance, bursting, density as well as oil absorption properties of the final product and the raw materials used, i.e. coir dust, cotton and jute, were determined as per American Society for Testing and Materials (ASTM) and Indian Standard (IS) methods. The OAM and the raw materials were also characterized by FTIR, XRD, TGA and SEM.

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Chapter 2

Objectives and experimental outlines

2.1 Objectives:

1. Study on the characterisation of coir pith such as calorific value, moisture content, ash content, cellulose content, lignin content, ph value, alkali solubility (1 % NaOH solution), cold water solubility, hot water solubility, bulk density, porosity, water holding capacity, Swelling % etc according to standard methods (TAPPI, 1980; AOAC, 1975).
2. Study different Physico-chemical treatments of coir pith as an absorbent to be used for prevention of oil spillage effect with reference to NE oil industries.
3. For improving hydrophobicity and oleophilic character of coir pith, Physico-chemical treatments such as heating at relative low temperature, acid treatment, alkali treatment, acetylation and acylation treatment were done and absorption in Water and oil were tested as per ASTM method. The properties of treated coir pith were characterized by FTIR and SEM.
4. Design and development of coir dust sorbent in the shape of membrane/ blanket/ block/ granules/ pad/ Sheet to deal with oil spillage.
5. Mechanical properties and oil sorption capacity of the developed Mat/ Sheet/ granules/ bags were done. Also comparison between various oil absorbent and oils were done and sent for field trial.
6. Study on possibilities of re-use & disposal of coir dust sorbent.

The whole research work of the project has been conducted at CSIR-North East Institute of Science and Technology

2.2 Experimental Outlines:

The physico-chemical properties of coir dust were evaluated as per TAPPI Standard Test Method and ASTM method. The Coir dust were suitably treated (i) chemically, (ii) physically and (iii) a suitably combination with other natural organic waste such as cotton waste and Jute waste thereof and the effect of such treatment on properties of coir dust were studied and compared with the untreated Coir dust. The downsizing of coir dust was done mechanically to generate particles of variant sizes which were subsequently separated into different sizes (mesh sizes) by screening process (sieve shaking). The composite boards were prepared by mixing of coir dust in the form of particles with binder under conditions of hydraulic hot pressing. The composite boards were characterized in terms of physical properties (density, water absorbency, oil absorbency, thickness etc.) and mechanical properties (tensile strength, breaking load, etc.). The process of composite board making was studied via examination of influence of different process variables (weight ratio of particle to time, temperature, pressure and binders (chemical agent)) on the properties of composite boards. The usefulness of the composite boards as oil absorbent was tested. Similarly the composite Oil absorbent Mat (OAM) has been developed using coir waste material, such that soil pollution from oil seepage into the soil from coir dust may be minimized. OAM is made from coir dust (CD) in combination with commonly available natural fibre i.e. cotton waste (CW) and jute waste (JW) in the ratios of 80:20, 85:15, 90:10 respectively. Various mechanical properties of the product, for instance oil absorption, double fold, bursting strength, ultimate tensile strength (UTS) etc. were determined along with thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). CD, CW and JW were characterized by FTIR, XRD, TGA and SEM analysis.

Chapter 3

Materials and Methods

3.1 Collection of coir pith and crude oil:

3.1.1 Collection of coir pith from different coir industry:

The coir pith was purchased from Coir Industry, Sonitpur (Biswanath Chariali source), Assam and was supplied by Amrit Organics, Duliajan Assam. Coir pith samples from Nalbari source, Assam have also been purchased for a comparative study. After collection, the moisture content of the Coir dust was determined in a laboratory moisture meter. The Coir dust (CD) were screened and then washed properly with cold fresh water and sun dried. The dried CD were packed in polythene bags and kept for subsequent study.

3.1.2 Collection of Crude oil samples:

Collection of crude oil samples were from

- a) Oil India Ltd., Duliajan, Assam and
- b) Pumping Station (PS-3), Oil India Ltd., Charigaon, Jorhat, Assam.

3.2 Proximate Chemical Analysis/ characterisation of Coir dust:

3.2.1 Determination of Ash Content:

According to the TAPPI standard method T-211 cm-86 (TAPPI, 1980), Coconut coir dust was air dried for 24hrs in oven at 60°C to constant weight. Coir dust (1g) was weighed in a Platinum crucible and then the crucibles were put into a muffle furnace at 575±25°C. After ignition for 4 hours, the crucible (with washed and unwashed sample) were cooled slightly and placed in desiccators. When it cooled to room

temperature, weighed the ignited crucible on an analytical balance to the nearest 0.1 mg. The results were expressed as % of the moisture free coir dust as follows:

$$\text{Ash content (\%)} = A/B * 100$$

Where, A= OD weight of Ash in g.

B= OD weight of the test specimen in g.

3.2.2 Determination of calorific value of coir dust:

The calorific value of coir dust was determined by taking two samples of coir dust (washed and unwashed) of two sources in an Automatic Bomb Calorimeter (Leco, AC-350, Model No. : 603-300-100).

3.2.3 Determination of Moisture Content in Coir Dust:

The unwashed coir dust (1g) was measured into a pre-weighed porcelain dish with cover and weighed. The coir dust and the container with lid open was placed in an oven at 60⁰ C for 5 hours. Further drying was carried out at 80⁰ C for 3 hours. The container with the lid was allowed to cool and reweighed with minimum exposure to atmosphere. The last drying was repeated for another 3 hours till a constant weight was obtained. ^[47] Moisture content determined according to the formula:

$$\text{Moisture Content (\%)} = [(W_1 - W_2) / W_1] * 100,$$

Where, W₁ is the initial weight of coir dust before drying and W₂ is the final weight of coir dust after drying.

3.2.4 Solubility in Cold Water:

Solubility of coir dust in water is determined by the TAPPI standard method T- 207 m-54 (TAPPI, 1980). 1 g of coir sample is digested at room temperature with 300 ml of distilled water with frequent stirring for 48 h. The mixture is then filtered and residue is washed with cold distilled water and then dried at 100⁰ C \pm 5 °C and

weighed in a stopper weighing bottle to constant weight. The loss in weight of the substance is calculated in Percentage as cold water soluble material.

$$\text{Cold water solubility (\%)} = [(A-B) * 100] / A$$

Where, A = Initial OD weight of the specimen in g.

B = OD weight of the specimen after extraction in g.

3.2.5 Solubility in Hot Water:

According to TAPPI standard method, T-207 m-54 (TAPPI, 1980), 1 g of coir sample is digested at with 100 ml of distilled water in a water bath under reflux condenser for 3 h. The mixture is then filtered and residue is washed with hot distilled water and then dried at 100-105°C to constant weight. The loss in weight of the substance is calculated in Percentage as hot water soluble material.

$$\text{Hot water solubility (\%)} = [(A-B) * 100] / A$$

Where, A = Initial OD weight of the specimen in g.

B = OD weight of the specimen after extraction in g.

3.2.6 Solubility in Dilute Alkali:

According to TAPPI standard method, T-207 m-54 (TAPPI, 1980), Oven dried coir dust (1g) is stirred with 100ml of 1% solution of NaOH, in covered beaker, which is placed in a boiling water bath for exactly 1 h with intermediate stirring at 10, 15 and 25 minutes. The mixture is then filtered by suction on a tarred crucible and the residue is washed in succession with hot water, 50 ml of 10% acetic acid and again with hot water. The final residue is then dried at 100-105 °C and weighed. The result is expressed as percentage on the oven dry weight of coir dust.

$$\text{Dilute Alkali solubility (\%)} = [(A-B) * 100] / A$$

Where, A = Initial OD weight of the specimen in g.

B = OD weight of the specimen after extraction in g.

3.2.7 Determination of cellulose content:

2 g of coir dust was taken in a conical flask; 10 ml of bleach (Sodium hypochlorite) is added in 90 ml of deionised water into the conical flask. The mixture was boiled until sample is bleached. It is then filtered, washed and dried at 80°C. To the bleached sample, 100 ml of 24 % aqueous NaOH is added and boiled for 1 hr to remove hemicelluloses. Then residue was filtered, washed and dried at 80 °C for 2-3 hours for getting constant weight for calculation. Calculation was made following the relation given below.

$$\text{Cellulose content (\%)} = A * 100 / B$$

Where, A = OD weight of cellulose in g.

B = OD weight of the test specimen taken in g.

3.2.8 Determination of Acid insoluble Lignin content:

According to TAPPI standard method T-13 m-54 (1980) 1g of the sample was taken, mixed with 15 ml of 72 % H₂SO₄ at 20 ± 1°C and kept in a water bath for 2 hr maintaining constant temperature 20 ± 1°C. Transferred the material to a beaker and added water so that total volume becomes 575 ml. Boiled the Solution for 4 hrs in a round bottom flask with 1m long glass pipe joint so that the vapours are cooled in pipe and come down and there is no loss. Keep the round bottom flask for overnight to settle the insoluble material. The final residue was then filtered, washed, dried at 105 ± 3°C and weighed. The result was expressed as percentage as follows.

$$\text{Lignin content (\%)} = A * 100 / B$$

Where, A = OD weight of acid insoluble lignin in g.

B = OD weight of the test specimen taken in g.

3.2.9 Determination of pH of coir dust in water:

1 g of dry coir dust (washed and unwashed each) was added to 30 ml tap water and allows soaking for 15 minutes. After that squeeze the coir dust from water and measure the pH of the each solution with pen type pH meter. It was found that pH for washed coir dust was 6.64 and unwashed was 6.90.

3.3 Physical properties of Coir dust:

3.3.1 Determination of percent swelling of coir dust in water:

Percent swelling was determine gravimetrically by swelling a sample of coir dust in deionised water and determine weight of the swollen sample and the weight of the corresponding dry sample. Coir dust 1 g was taken in 100 ml of deionised water in a conical flask for 1 hour. The weight of the swollen sample was determined and weight of the dried sample determined.

$$\% \text{ Swelling} = A * 100 / B$$

Where, A = weight of the swollen coir dust

B = weight of the dry coir dust

3.3.2 Determination of Bulk Density of coir dust:

We take a beaker and determine its volume V and its weight W₁. Then we fill the beaker with coir dust and take its weight W₂.

$$\text{Bulk density} = (W_2 - W_1) / V$$

3.3.3 Determination of Total porosity, Aeration porosity and Water holding porosity of Coir dust:

In order to determine the porosity, a plastic cylindrical container of 250 ml capacity was taken. The bottom of the container was pierced with a fine needle so as to have 10 holes uniformly distributed at the bottom of it. The 10 holes were then closed with a waterproof adhesive tape. Then the container was filled with the coir dust by gently tapping it till the coir dust fills the 250 ml mark. The water was slowly dripped over the coir pith so that the coir pith was completely drenched and saturated. This process took several hours. The total volume of water added (A) in ml was recorded. Then the container was placed over a water proof pan and the adhesive tape at the bottom was removed so as to drain the water on the pan. Then the drain water (B) in ml was measured.

$$\text{Total porosity \%} = A * 100 / 250$$

$$\text{Aeration Porosity \%} = B * 100 / 250$$

$$\text{Water holding porosity} = \text{Total porosity} - \text{Aeration porosity}$$

3.3.4 Determination of percent weight of graded Coir dust:

1000 g of Coir dust sample is taken for sieve analysis to assess the particle size distribution. From the distribution it was seen that $> 1.18 \text{ mm}$ to $< 600 \mu\text{m}$ size particles were present in highest %. Also particle size between 2.36 mm to 1.18 mm and $600 \mu\text{m}$ to $300 \mu\text{m}$ were mostly presents in the graded coir dust.

3.4 Physico-Chemical Treatment of coir dust:

3.4.1 Heat treatment of coir dust at relatively low temperature:

Heat treatment was done by heating 15 g of coir dust at 150°C for 17 minutes in hot air oven. This treatment of coir dust was done to improve its hydrophobicity.^[45]

After that it was exposed to water and oil to determine the hydrophobic and oil sorption capacity.

3.4.2 Acetylation treatment of coir dust:

1 g pre-treated (washed and dried) coir pith of 600 μm was placed in a 250 ml R.B. flask containing 20 ml of acetic anhydride solution and 0.05 g DMAP. The flask was heated at 120 $^{\circ}\text{C}$ for 30 minutes with reflux condenser fitted. After that the coir dust was washed by ethanol and acetone to remove the unreacted acetic anhydride and acetic acid by product. The product was then dried at 50 $^{\circ}\text{C}$ for 16 hours in hot air oven.^[45] Acetylated coir product was carried out for absorption test.

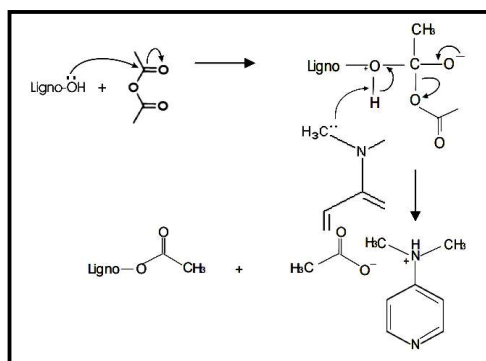


Figure: The mechanism of Acetylation reaction on Lignocelluloses

3.4.3 Acid treatment of coir dust:

We have done acid treatment of 600 μm coir dust particles by mineral acid. Acid treated coir pith was carried out for absorption test.

3.4.4 Alkali treatment of coir dust:

10 g of washed and dried coir dust was taken in a R.B. flask and added 300 ml of 5 % NaOH to it. Heat the mixture at 100 $^{\circ}\text{C}$ for 3 hours. After that the mixture was cool for whole night. Washed it properly with distilled water and dried for 5 hours at

60 °C in hot air oven. After that weight of the alkali treated coir dust was taken and it was found that 85.22 % of coir dust was digested. Alkali treated coir dust was exposed to oil and water for sorption test.

3.4.5 Esterification of coir dust with fatty acid chloride (Acylation treatment):

Bio sorbent (Coir dust) Preparation:

The coconut coir raw material used in this study originated from Biswanath Chariali source, Assam. The raw coir pith was ground into small and short shapes in a high speed grinder, and the visible dust/impurities were removed by washing and boiling with distilled water. Following this, the coir pith was washed several times with acetone before drying in the oven at 60 °C overnight. To ensure the freshness of the raw materials, no further treatment was carried out. ^[46]

Esterification Process:

The Esterification reaction of coir was carried out in a reflux setup maintained under a fume hood. For that, approximately 5 g of coir pith was weighed and placed in a 250 ml two necked round bottom flask. To this, 100 ml of 1% (v/v) N-bromosuccinimide (NBS) catalyst prepared in a DMAc / LiCl solvent system was added and heated up to 100 °C with stirring. To this mixture, 10 % (v/v) concentration of Oleoyl chloride was added drop-wise, and the reaction was allowed to proceed continuously for 4 hours under the same conditions of temperature and stirring. After the period during which the Esterification process was completed, the Acylated coir dust was filtered and washed with a series of solvents including toluene, ethanol, water, and acetone to remove any unreacted oleoyl chloride and unwanted by-products. Then the coir dust was dried to a constant weight in an oven at 70 °C.^[46] The same synthetic process was repeated

for 20 % & 30 % oleoyl chloride are done. Absorption test in Crude oil were done for the same.

3.5 FTIR study of treated coir dust:

The coir dust was evaluated by FT-IR. FT-IR studies were conducted by using IR affinity-1, Shimadzu, Japan; FT-IR Spectrophotometer.

3.6 Composite board making:

Boards were made in the laboratory using untreated and chemically treated Coir dust and different binders (LDPE, Starch & Foaming Agent). We make the blocks of coir dust from two sources (Biswanath Chariali coir dust & Nalbari Coir dust) mixed with each binders in such a way that these were uniformly distributed all around the Coir dust mass. The mixture was put into the wooden mould size 30 × 30 cm and hydraulic hot pressed at 140 ± 2 °C for 20-30 minutes and at 2 kg/cm² pressure. After that, the pressure was released from the hydraulic hot press and the board was kept for some time in open air for conditioning. The properties of the boards made from each treated coir dust and binders were studied. The desirable characteristic in the block was stability and high absorption rate.

3.7 Testing of Composite board Samples (Mechanical & Oil Sorption):

Tensile strength of composite specimens was analyzed at room temperature and 55% RH using Universal Testing Machine (UTM) INSTRON Make, Model 5594. Ultimate tensile strength, maximum load, tensile modulus values were calculated by the software Merlin software version V22054. The values of elongation at break were calculated using equation:

$$\text{Elongation at break (\%)} = \text{Tensile Strength/Tensile Modulus} \times 100$$

For determination of Modulus of Rupture (MOR), 3 point flexural test attachment was used. The MOR was then calculated and expressed in N/mm² by equation:

$$R = 3PL / 2bd^2$$

Where,

P - Maximum load in kg

L - Length of span in cm

b - Width of specimen in cm

d - Depth of specimen in cm

Absorption rate of composite coir dust board were checked in crude oil by cutting the composite boards into small blocks in different interval of times. Then calculate the weight gained by the blocks and graphs were representing with respect to time vs. weight gained.

3.8 Development of Coir dust Bag for Absorption Test in crude oil:

To use Coir dust practically for prevention of oil spillage in the oil fields and other affected areas we have prepared coir pith bags/ pouches. We make bags of markin cloth for containing treated coir pith and check its absorption rate in crude oil.

3.9 Development of coir dust handmade paper (round) for absorption test in crude oil:

Procedure: Coir dust was taken and beat it in the beating machine for 2-3 hours. Similarly cotton waste were beat in the same process to make pulp. After that collected the samples and mixed them in specific ratio (10% & 20% Cotton waste) and make sheet of the same in the Sheet form machine. Then the sheet formed was pressed in the screw pressed machine such that it releases the water from the pulp

sheet. After that dried it for the day and exposed to oil and water to check its sorption capacity

3.10 Development of coir dust various treated handmade paper for absorption test in crude oil:

We made various treated coir dust composite handmade paper. Those were Oven dried coir dust paper; Hot plate press handmade coir pith paper; Oil treated handmade coir pith paper and Latea treated handmade coir pith paper. Expose those papers to oil and water for absorption.

3.11 Development of a high oil absorbent mat from coir dust and evaluation of their Physico-chemical properties:

Coir dusts were collected from North East Coconut Products (P) Ltd., Assam, India, which were initially screened to carry out the experimental work. Natural fibres i.e. cotton waste (hosiery cuttings) and jute waste (gunny bag cuttings) were collected from nearby local market of Jorhat, India and cut initially into the size ranged between 1.5-2.0 cm length to carry out all the experiments. All the chemicals used in the experimental work were procured from Spectrochem, India and used without any purification.

Proximate chemical constituents of coir dust, jute and cotton waste were carried out using the analytical method suggested by Technical Association of Pulp and Paper Industry (TAPPI, USA). For determination of chemical constituents, the CD powder, jute and cotton waste samples were cut into 1.5-2.0 cm length and dried in oven for a period of 6-7 h at 90 ± 5 °C and then powdered in a Wiley mill. The powdered fraction was screened with 40 and 60 BSS mesh and the fraction passed through 40 BSS mesh and retained on 60 BSS mesh (+40 -60) was taken for constitutional analysis.

Lignin content of plant materials was determined by TAPPI, T-222 om-83 and cellulose content by a method of suggested by S. K. Thimmaiah [23]. FTIR spectra (4000-500 cm⁻¹) were recorded in a Shimadzu IR Affinity-1 spectrophotometer on KBr discs. Thermal gravimetric were carried out using TA Instrument (SDT Q600). Under a nitrogen environment flow of 100 ml/min, the samples were heated from 20 °C to 700 °C at a heating rate of 10 °C/min. Scanning Electron Microscopy of CD, NF and OAM were carried by Leo 1430 vp operated at 3 KV on gold coated sample and images were collected at different magnifications. Powder XRD diffractions were carried out on a Rigaku, Ultima IV X-ray diffractometer from 2-80° 2θ, using CuKα source (λ=1.54056 Å). The crystallinity index (CI) was analysed using Equation 1, where I₀₀₂ is the maximum intensity of the I₀₀₂ lattice reflection and I₁₀₁ is the maximum intensity of X-ray scattering broad band, because of amorphous region of the sample [24].

$$CI (\%) = \frac{I_{002} - I_{101}}{I_{002}} \times 100 \quad \dots\dots\dots 1$$

Tensile strength of CD, NF and OAM were determined at 25 °C and 55 % RH using Universal Testing Machine, Make TWI, Model TUTE 10T and values were also calculated by the UTM10 DCCP TWI LCD software version SP2 PACK 04/13. According to ASTM D6182 test method, folding endurance strength was also determined using double fold tester, MIT type machine, Model UEC-1007-C. With the help of Bursting strength tester, Model UEC-1010-B1, bursting strength carried out according to ASTM D2207 test method. Oil absorption test for the oil absorbent mat using different oils (Crude Oil, Diesel Oil, Hydraulic Oil, Transformer Oil, Mobil Oil) at 20- 30 °C was conducted following IS specification [25].

3.11.1 Preparation of Oil Absorbent Mat (OAM) sample:

Coir dust, Cotton waste i.e. hosiery cuttings and jute waste i.e. waste gunny bag cuttings were samples were beating separately with a laboratory beater for 3 h using deionised water at 1.5 % uniformity. Two separate stocks prepared from coir dust, cotton waste/ jute waste were supplemented with 4 % guar gum and 1 % alum during stock preparation. Absorbing mat were made in the handmade vat, keeping coir dust waste to jute/cotton at 80:20, 85:15, 90:10 ratio, followed by screw press machine to eliminate the surplus water from the wet mat/pad and then drying of mats.

Chapter 4

Results and Discussions

4.1 Proximate Chemical Analysis/ Characterisation of coir dust:

The results of proximate chemical analysis/ characterisation of coir dust are shown in Table 1. The study shows that there are lots of soluble materials in Raw Coir dust. Cellulose content were found to be 35.90 % (Nalbari Source coir dust) and 36.56 % (Biswanath Chariali Source coir dust), lignin content were 44% and 44.9 % for Nalbari Source & Biswanath Chariali Source coir dust respectively.

Table 1: Proximate chemical analysis of the coir dust

<i>Particulars</i>	<i>Biswanath Chariali source (%)</i>	<i>Nalbari source (%)</i>
Ash Content	13.89	13.97
Calorific Value	Washed: 3820, Unwashed: 3850	Washed: 3825, Unwashed: 3857
Moisture Content	24.9	25.3
Solubility in Cold Water	10.5	11.2
Solubility in Hot Water	13.9	14.3
Solubility in 1% NaOH	17	17.2
Cellulose Content	36.56	35.90
Lignin Content	44.9	44
pH	6.64	6.65

4.2 Physical properties of Coir dust:

The results for physical properties of coir dust were shown in Table 2 & Table 3. The study shows the bulk density, porosity, swelling % (Table 2) and distribution of particles size of coir dust (Table 3)

Table: 2

<i>Particulars</i>	<i>Biswanath Chariali source</i>	<i>Nalbari source</i>
Bulk Density (Kg/ cm ³)	156.393	155.202
Total Porosity (%)	74.4	75.75
Aeration Porosity (%)	18.8	20.05
Water Holding Capacity (%)	55.6	55.7
Swelling %	65.88	-

Table: 3 Determination of percent weight of graded Coir dust

<i>Average particle Size (mm)</i>	<i>Weight of coir dust (gm)</i>	<i>Weight %</i>
+10	30	3.0
-10 to +5.6	72	7.2
-5.6 to +4.75	70	7.0
-4.75 to 2.36	110	11.0
-2.36 to +1.18	196	19.6
-1.18 to +0.600	236	23.6
-0.600 to +0.300	222	22.2
-0.300	64	6.4

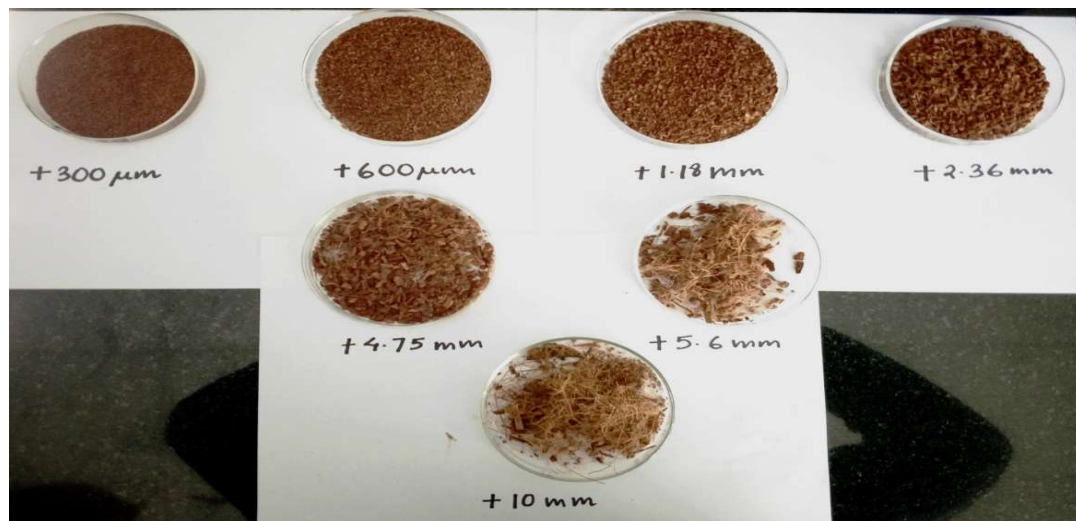


Figure 1- Particle size distribution of coir sample

4.3 Determination of viscosity of crude oil:

The oil sample is placed into a glass capillary U-tube and the sample is drawn through the tube using suction until it reaches the start position indicated on the tube's side. The suction is then released, allowing the sample to flow back through the tube under gravity. The narrow capillary section of the tube controls the oil's flow rate; more viscous grades of oil take longer time to flow than thinner grades of oil.

Table: 4

Rheology of raw crude	Sample 1			Sample 2			Sample3		
Temp °C	18	21	24	21	24	27	24	27	30
PV (C_p)	30	29	20	24	20	10	22	18	12
YV (dynes/cm²)	20	12	8	10	8	2	8	6	2

4.4 Determination of Sorption Capacity of graded coir dust in water & crude oil:

The sorption capacity of coir dust (graded) was checked both in water and crude oil. We made two samples of all graded coir dust; one was raw and another was heat treated at relatively low temperature. On heat treatment, the dried coir dust was kept in hot air oven at 150 °C for 17 minutes. The water sorption capacity decreases in heated coir dust due to melting of lipophylic extractive compounds in the pores of coir dust. The lipophylic extractive compounds like wax and gum melted and made hydrophobic thin film on the surface of the pore. Heat treatment also reduces the weight of the coir dust. The weight reducing of heated coir dust might be caused of the lack of water content and volatile extractive content. The results of the sorption of water and crude oil were shown in the table 5 and 6 respectively. Also graded coir dust vs. weights gained by coir dust after sorption of water and oil graphs were shown.

Table: 5 Weight gained by coir dust after absorption of water

<i>Average particle Size (mm) of coir</i>	<i>Weight of Untreated coir dust sample after absorption of water (gm/gm of coir dust)</i>	<i>Weight of Treated coir dust sample after absorption of water (gm/gm of coir dust)</i>
Sample (as received)	5.1564	4.5372
10	2.6039	2.0802
-10 to +5.6	3.0299	2.7555
-5.6 to +4.75	3.1505	3.0869
-4.75 to +2.36	3.8968	3.5293
-2.36 to +1.18	5.1148	4.8952
-1.18 to +0.600	5.9814	5.1521
-0.600 to +0.300	7.0810	6.6627

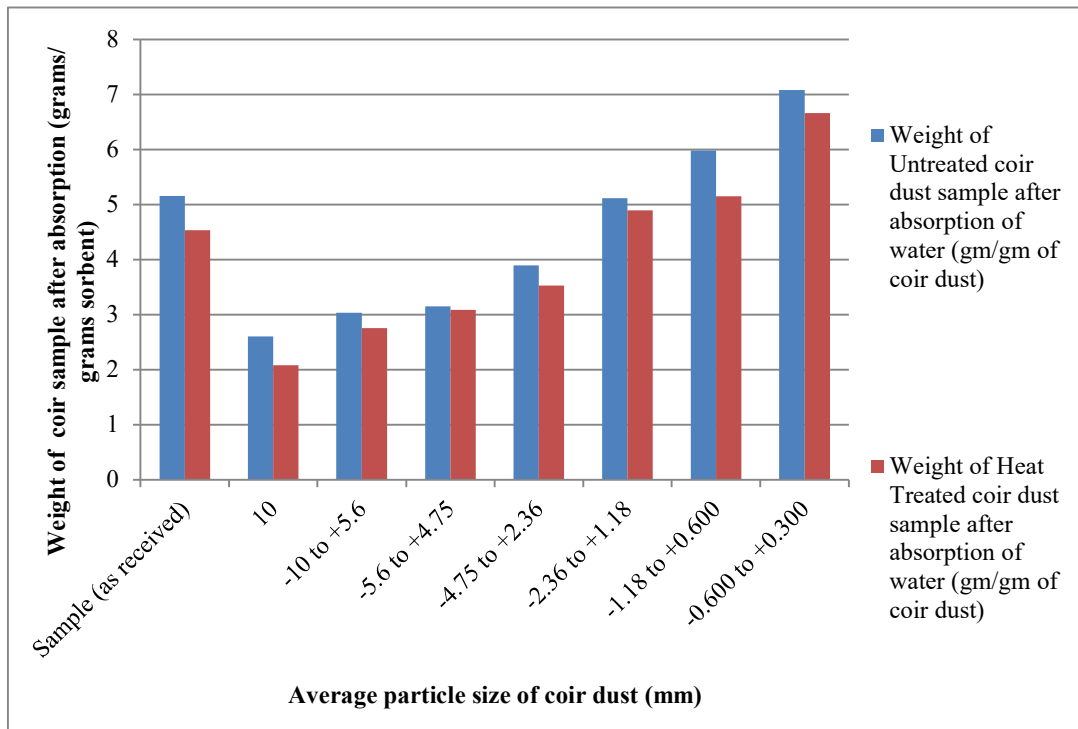


Figure 2- Absorption test of coir dust in Water

From the sorption test it was seen that water sorption was little decrease in heat treated coir dust. The above chart shows the change in absorption of coir dust in different particle size.

Table: 6 Weight gained by coir dust (CD) after absorption of crude oil

Average particle Size (mm)	Weight of Untreated CD sample after absorption of crude oil (gm/gm of CD)	Weight of Treated CD sample after absorption of crude oil (gm/gm of CD)
Sample (as received)	4.1772	4.2219
+10	1.5808	1.6184
-10 to +5.6	1.8124	1.8668
-5.6 to +4.75	2.3712	2.9084
-4.75 to +2.36	2.8410	3.7829
-2.36 to +1.18	3.5098	3.8019
-1.18 to +0.600	5.0289	5.8314
-0.600 to +0.300	5.9702	6.4438

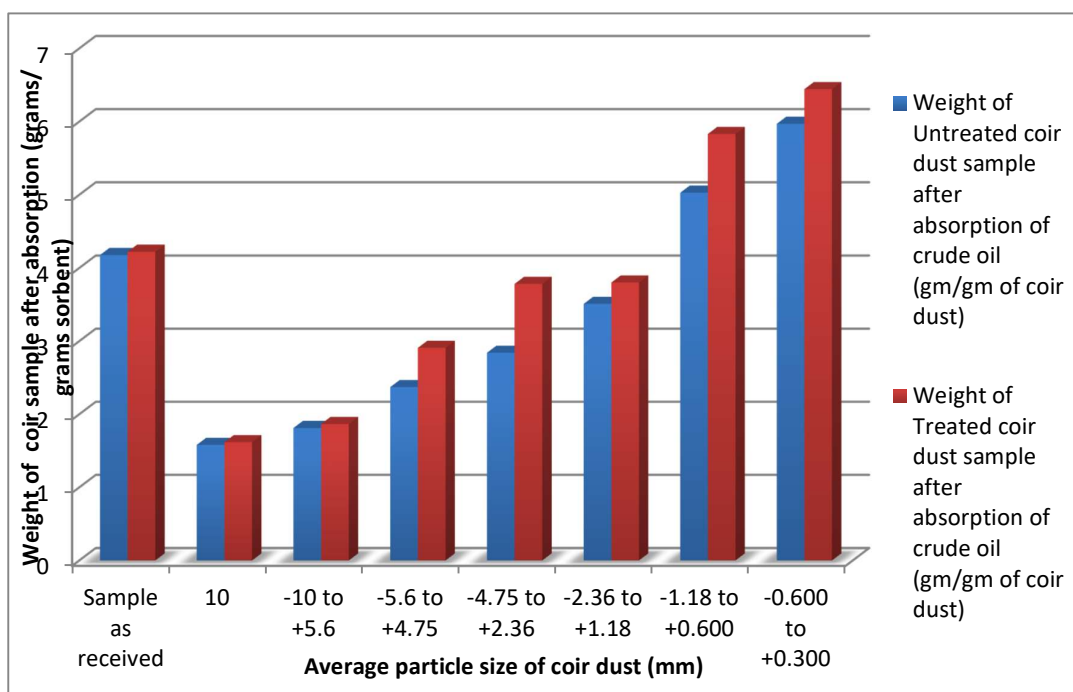


Figure 3- Absorption test of coir dust in crude oil

From these tests it was seen that in heat treatment there was increase in sorption of crude oil. It indicates that the hydrophobic character of coir dust increase since the lingo-cellulosic O-H bond reduces. From the above charts it was seen that absorption rate depends upon the particle sizes of the coir dust.

4.5 Sorption of water by the saw dust and mixture of saw dust and coir dust sample:

The saw dust have been heated at 70 °C for 30 minutes.

Table: 7

<i>Sample</i>	<i>Weight of the sample before absorption in gm</i>	<i>Weight of the sample after absorption in gm</i>
Saw dust	5.0055	31.01581
Coir + saw dust (1:1)	5 (2.5+2.5)	35.3377

4.6 Physico-Chemical Treatment of coir dust:

The Physico-chemical treatments were done to improve hydrophobicity and oil sorption capacity of coir dust. Hydrophobicity of coir dust was approached by using water sorption capacity of coir dust. Effects of various treatments onto coir dust were showed in Table 8 and Figure 4. Fig. 4 shows that the decrease of water sorption capacity on

Heated coir dust might be caused by melting of lypophylic-extractive compounds in the pores of coir dust. The lypophylic-extractive compounds, like wax and gum, melted and made a hydrophobic thin film on the surface of the pore. After heating at 150 °C for 17 minutes, the weight of heated coir dust decreased down. The weight reducing of heated coir dust might be caused of the lack of water content and volatile extractive content.

In Acetylation treatment, the decrease of water sorption capacity on acetylated coir dust related to the efficiency of acetylation process. The O-H groups of lignocellulose was reduced so the probability to form hydrogen bonding with water molecules decreased.

In acid treatment, the absorption of water and oil both increases simultaneously and in alkali treatment (Digestion treatment), it is seen that hydrophobic as well as oleophilic character improves and it absorbed 4.0869 g of water/ g sorbent and 8.0607 g crude oil/ g of sorbent. But in alkali treated (digested) coir dust the weight loss of coir dust is more as compared to other treatments.

Table: 8 Absorption of water and C. Oil of treated Coir Dust

<i>Coir dust (CD) Sample</i>	<i>Weight absorbed by CD by water absorption (gm/gm of sorbent)</i>	<i>Weight Absorbed by CD by crude oil absorption (gm/gm of sorbent)</i>
Raw	5.9814	5.0289
Heat treated	5.1521	5.8314
Acid treated	6.0750	6.6673
Acetylated	4.4339	6.7777
Alkali treated (Digested)	4.0869	8.0607

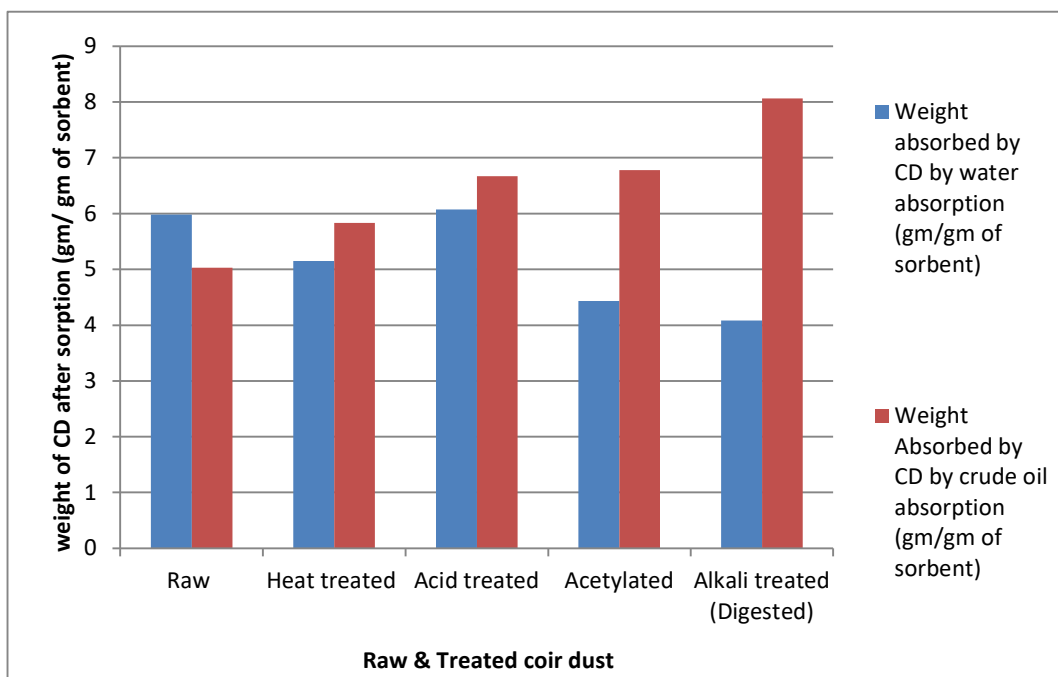


Figure 4 - Sorption test of raw and treated coir dust in water and C. oil

Esterified coconut coir dust was successfully done using oleoyl chloride by the replacement of hydroxyl groups of polymer backbone with that of acyl groups so as to achieve sufficient hydrophobic behaviour to interact highly with the similarly hydrophobic oil molecules.^[46] It was seen that with increase in concentration of oleoyl chloride from 10 % to 20 % and 30 % during esterification, the hydrophobic character of the formed coir dust-oleate was increased and also the oil sorption capacity was increased. When tested for oil adsorption studies, the oleoyl esterified coir dust (30%) was found to have better oil adsorption than the corresponding 10%, 20% oleoyl esterified coir dust and unmodified coir dust. The adsorption of crude oil was shown in Table 9 and figure 5. Based on the calculations, it was seen that the coir-oleate can serve as an alternative for oil spill due to its easy modification, abundance and biodegradability.

Table 9: Sorption of crude oil by raw and esterified coir dusts.

<i>Sample/Absorption</i>	<i>Weight absorbed by the coir dust after absorption of crude oil (gm/gm of sorbent)</i>
Raw coir dust	6.0310
Acylated coir dust (Esterified 10% Fatty Acid chloride)	9.6316
Acylated coir dust (Esterified 20% Fatty Acid chloride)	10.4612
Acylated coir dust (Esterified 30% Fatty Acid chloride)	11.3230

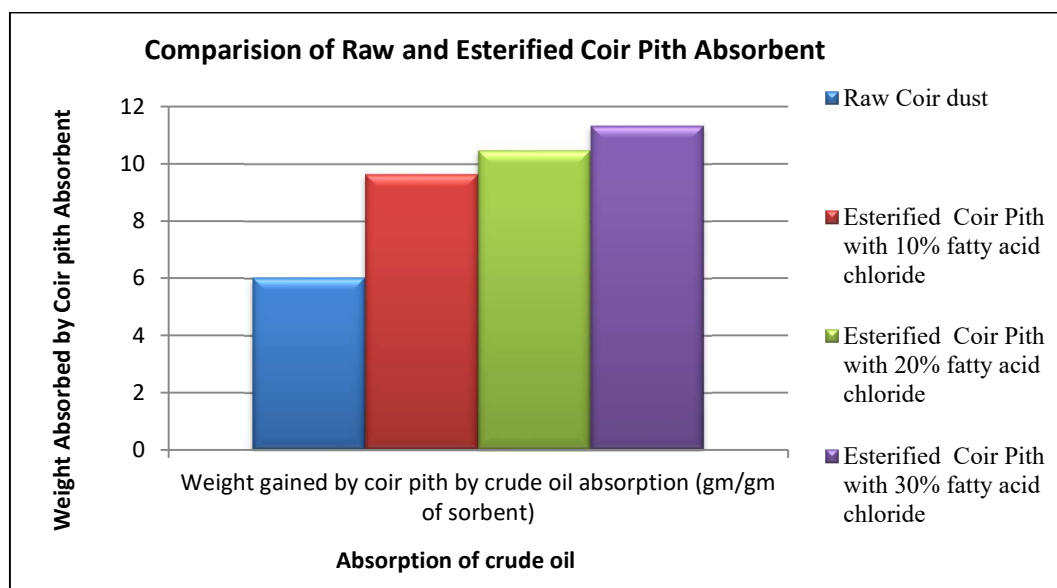
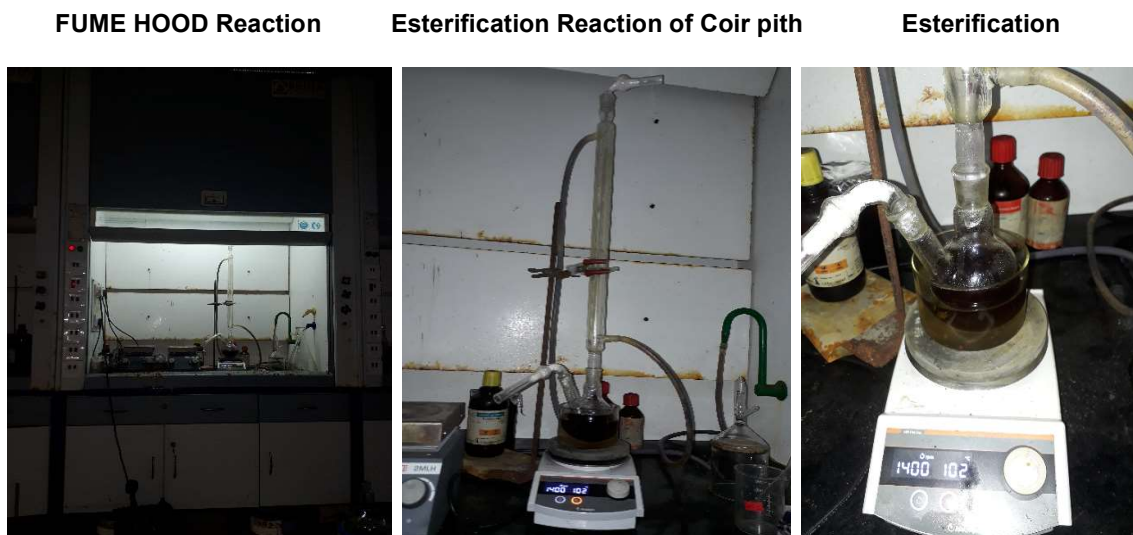


Figure 5 – Sorption of raw and esterified coir dust

Experimental Photos of Esterified Coir Dust



At the time of washing the Acylated Coir pith



Pre-treated Coir Pith (Boiled & Washed)



Esterified (Acylated) Coir Pith



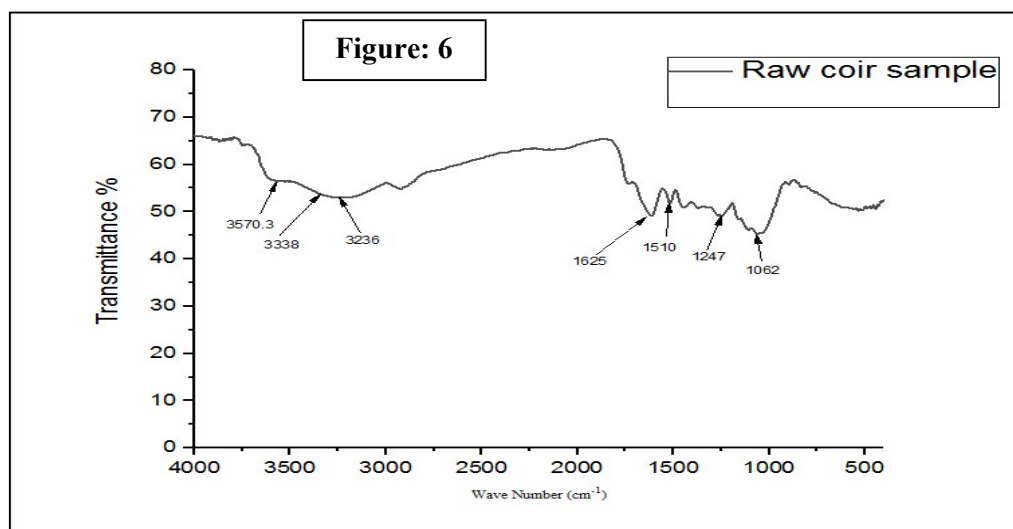
4.7 FT-IR spectra of Raw, Heat treated and Acetylated coir dust:

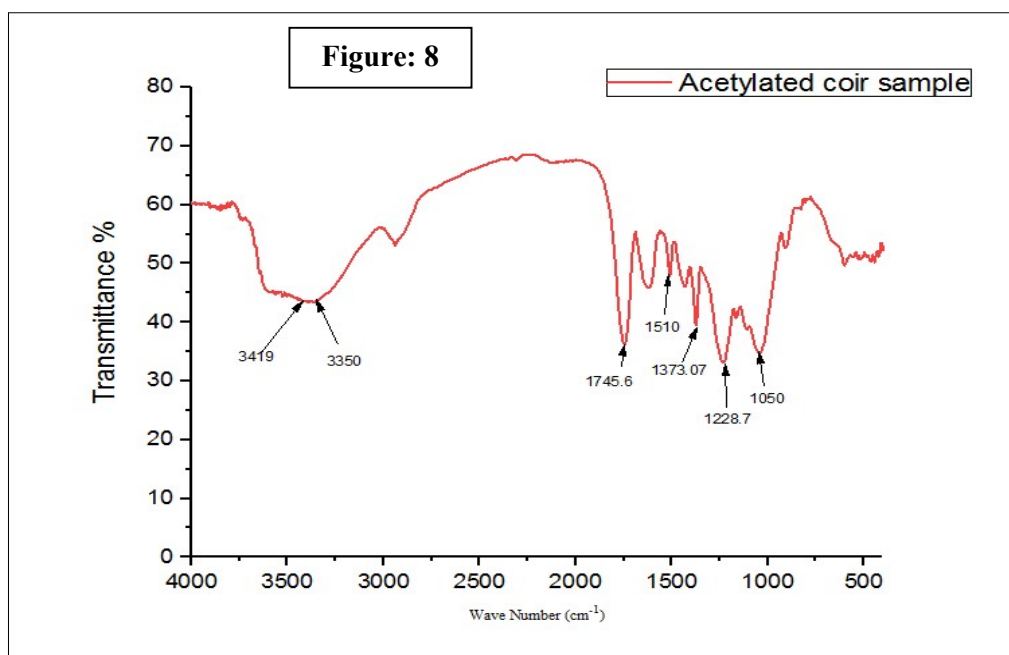
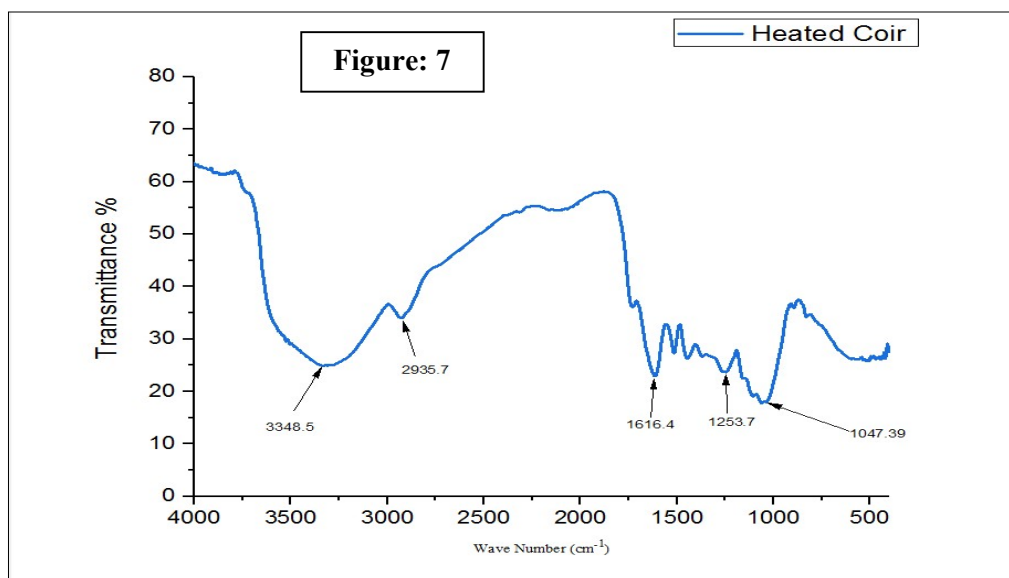
FT-IR spectra of the natural coir dust and the heated coir dust were showed in Figure 6 and Figure 7. Figure 6 and Figure 7 indicated no significant differences spectrum bands between unmodified coir dust and heated coir dust. The result indicated that heating treatment at 150 °C for 17 minutes didn't alter chemical structure on lignocelluloses of coir dust, only decreased the intensity of the O-H group absorption band at 3200-3300 cm⁻¹ in figure 7.

The FT-IR results indicate that coir dust has undergone modification by means of acetylation reaction (figure 8). The strong and sharp bands at 1745 cm^{-1} was attributed to absorption by C=O ester stretching, and at 1373.07 cm^{-1} was assigned to C-H stretching in -C-CH₃ group. Another strong band at 1228.7 cm^{-1} showed the C-O stretching in the acetyl group.

The decrease in the intensity of the O-H group absorption bands at 3419 cm^{-1} in spectrum of acetylation (figure 8) indicated that the acetylation was occurred to the O-H groups. The absence of absorption band at 1840 – 1760 cm^{-1} in spectrum of acetylation indicated that the acetylated coir dust was free of the unreacted acetic anhydride. The lack of absorption band at 1700 cm^{-1} stated that the acetylated coir dust was free of the byproduct of acetic acid also.

The other peaks 1510 cm^{-1} is for lignin & 1240 cm^{-1} for hemicelluloses.





4.8 Testing of Board making with the mixture of coir dust and binder:

Mechanical & Oil Sorption of Coir dust mixture with binder was shown in Table 10.



Figure 9- Coir dust Board of Nalbari & Biswanath Chariali Source of coir dust

Table 10: Mechanical and Physical Properties of composite Boards:

Sample	Density (g/cm ³)	Moisture content (%)	UTS (MPa)	MOR (MPa)	Breaking Load in tensile test (N)
Coir pith + LDPE	0.421	29.50	6.32 ± 0.12	12.51	885
Coir pith + Starch	0.445	33.40	4.16 ± 0.13	9.78	640
Coir pith + Starch (High Pressure)	0.681	28.10	4.86 ± 0.11	10.15	725
Coir pith + Foaming Agent	0.381	44.30	3.09 ± 0.14	6.35	620

Absorption of coir composite board in crude oil:

(i) Absorption of Coir block (Binder 1) in crude oil:

Make the coir pith block using binder 1 (LDPE). Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has

density **0.421 g/cm³** and weight of that piece was **12.7882 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 11 Weight gained by coir composite board after sorption of crude oil.

Time (in min)	15	30	45	75	105	135	235	335	435	565	3 days
Weight absorbe d by the coir block	17.337	18.571	19.183	20.145	21.115	22.273	25.023	26.860	27.923	28.077	30.168

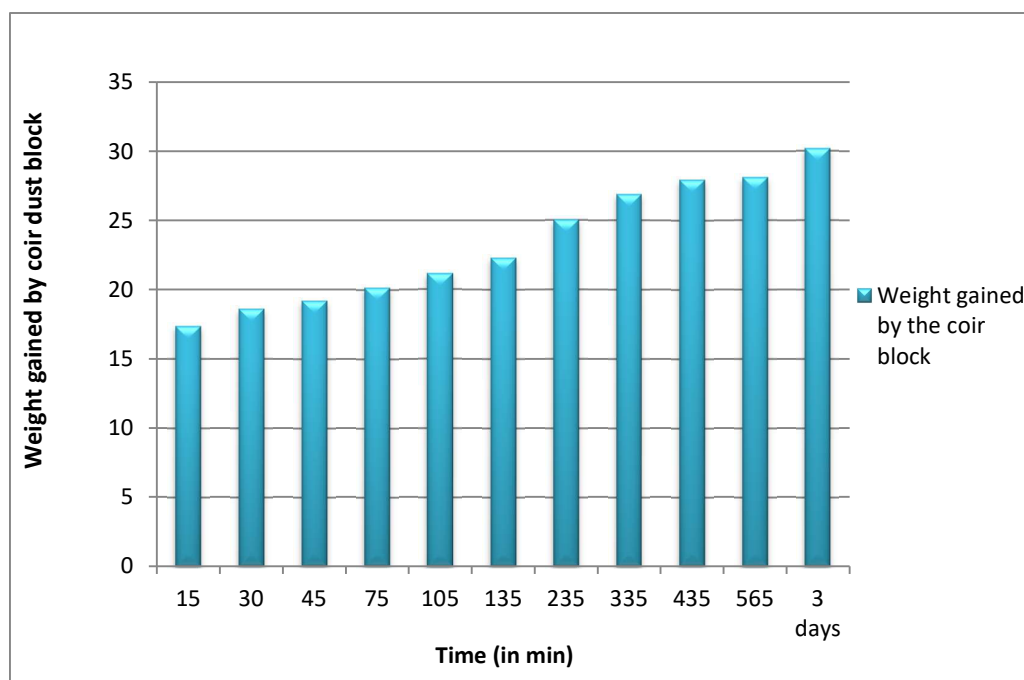


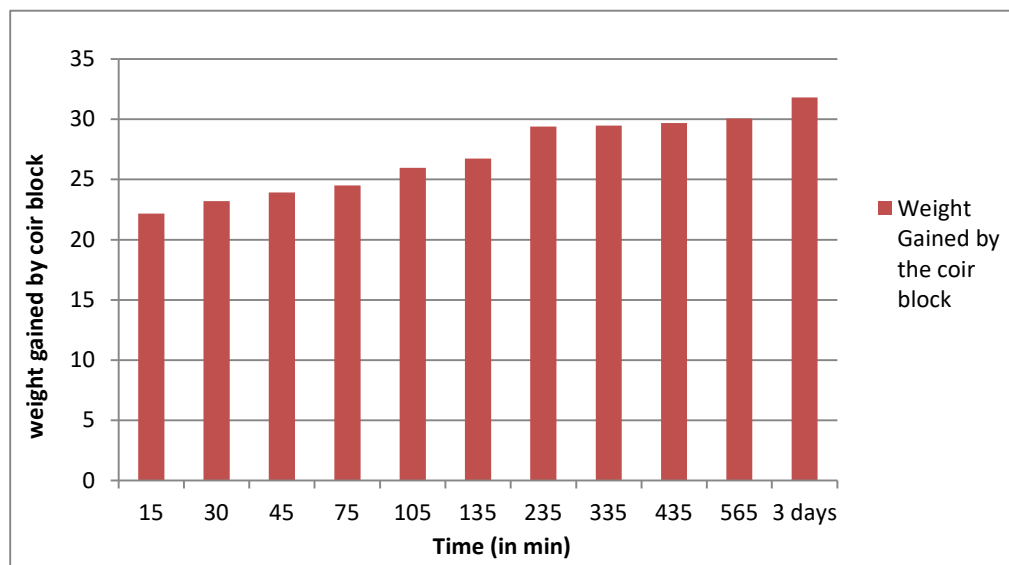
Figure 10: Absorption Rate vs. Time graph

(ii) Absorption of Coir block (Binder 2) in crude oil:

Make the coir pith block using binder 2 (Starch). Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.352 g/cm³** and weight of that piece was **14.9267 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 12 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	45	75	105	135	235	335	435	565	3 days
<i>Weight absorbed by the coir block</i>	22.166	23.200	23.901	24.494	25.971	26.734	29.394	29.451	29.682	30.052	31.801

**Figure 11: Absorption Rate vs. Time graph****(iii) Absorption of Coir block (Binder 2, High pressure) in crude oil:**

Make the coir pith block using binder 2 (Starch) with high pressure. Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.681 g/cm³** and weight of that piece was **13.7951 g**.

The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 13 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	45	75	105	135	235	335	435	565	3 days
<i>Weight absorbed by the coir block</i>	15.726	16.137	16.564	17.145	17.810	18.391	19.340	19.641	20.511	20.864	20.906

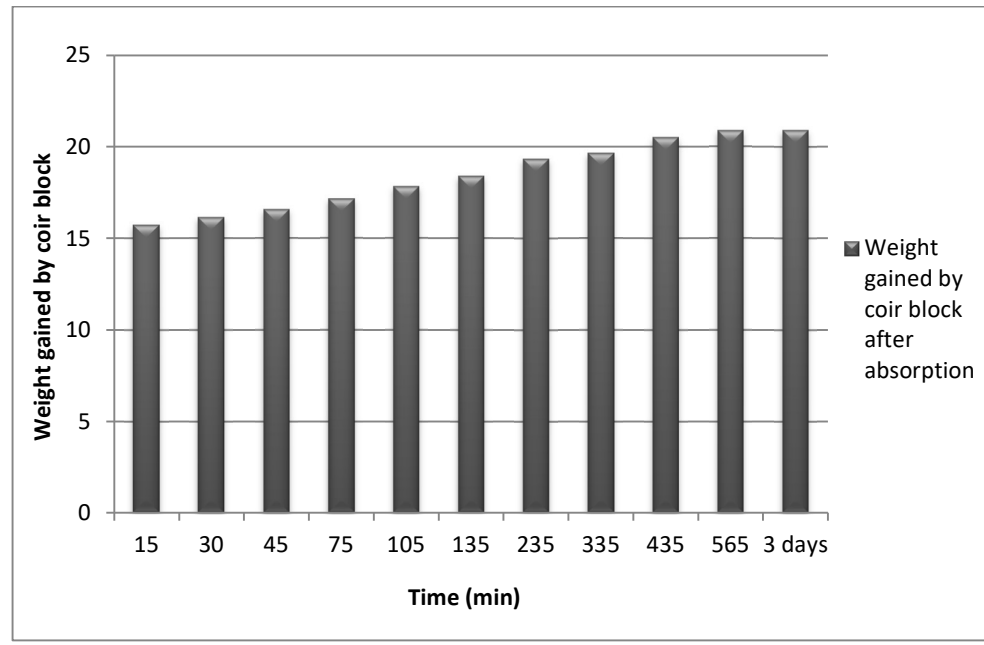


Figure 12: Absorption Rate vs. Time graph

(iv) Absorption of coir block (Binder 3, Biswanath Chariali source) in crude oil:

Make the coir pith block using binder 3 (Foaming Agent) to Biswanath Chariali source of Coir pith. Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.311 g/cm³** and weight of that piece was **14.9328 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 14 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	60	90	150	210	300	1 Day
<i>Weight absorbed by the coir block</i>	40.971	40.974	41.224	41.817	42.354	43.062	43.771	44.649

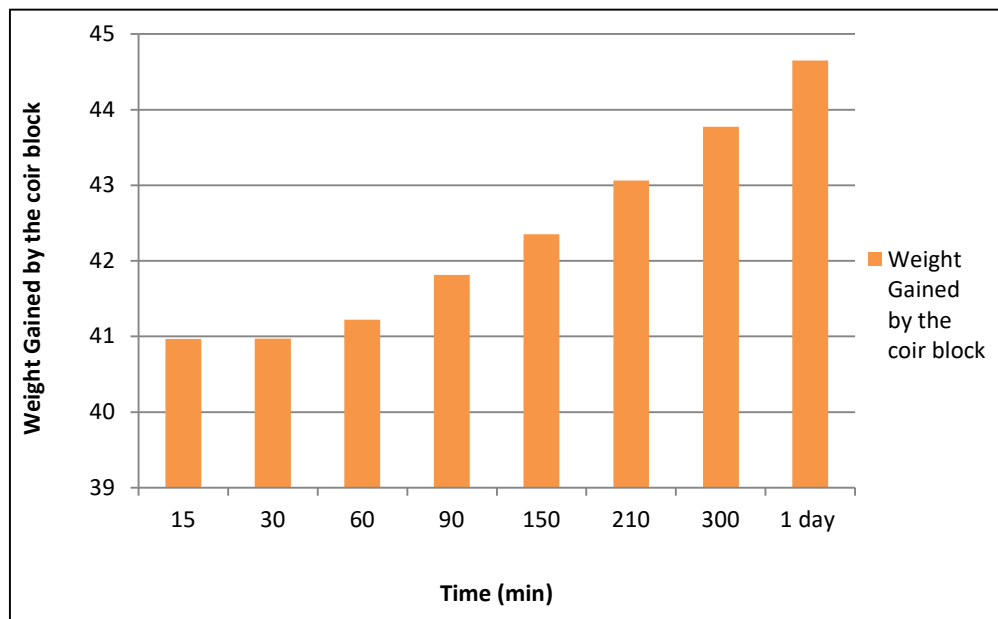


Figure 13: Absorption Rate vs. Time graph

(v) Absorption of coir block (Binder 3, Nalbari source) in crude oil:

Make the coir pith block using binder 3 (Foaming Agent) to Nalbari source of Coir pith. Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.381 g/cm³** and weight of that piece was **16.6160 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 15 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	60	90	150	210	300	1 Day
<i>Weight absorbed by the coir block</i>	30.480	32.445	33.420	34.175	35.292	35.953	36.566	37.959

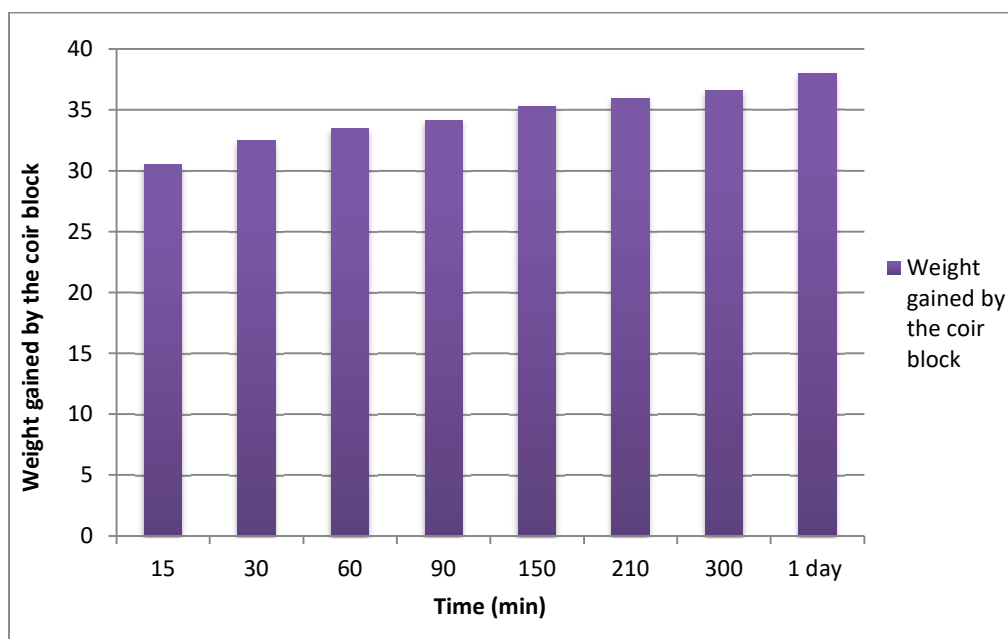
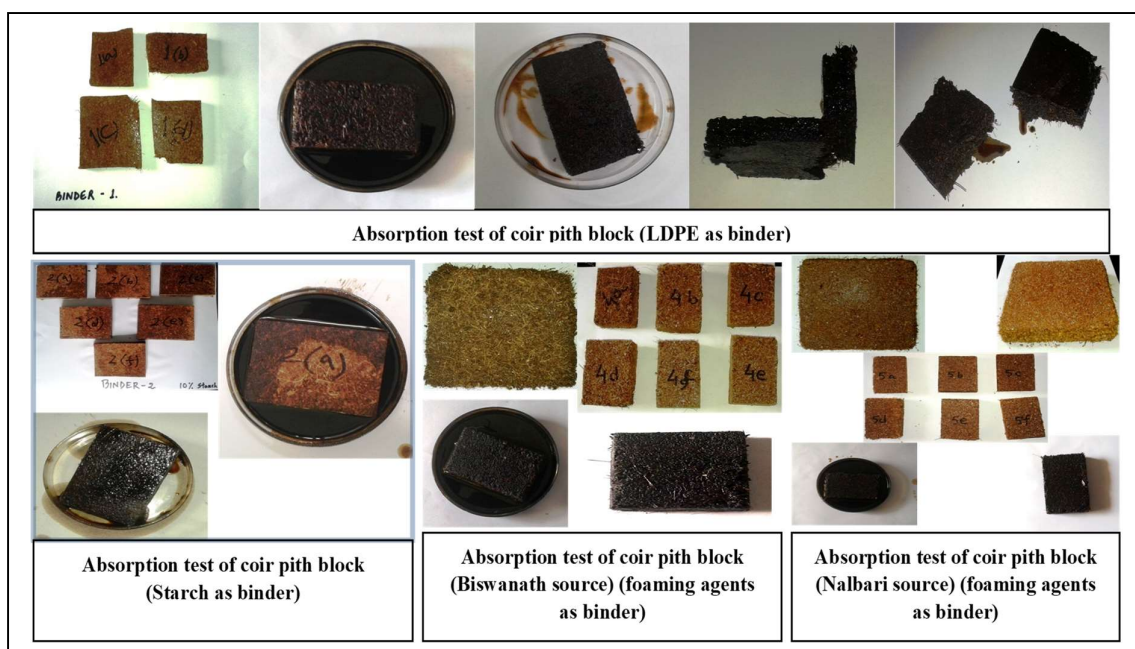


Figure 13: Absorption Rate vs. Time graph

The absorption rate of crude oil was better in coir pith block of Biswanath Chariali source (binder 3). It has fastest and highest absorptive capacity among all other coir pith block. Nalbari source of coir pith has better absorption rate with binder 1.

Experimental Photos



4.9 Testing of coir dust bags for absorption of crude oil:

To use coir practically for prevention of oil spillage in the oil fields and other affected areas we have prepared coir pith bags/ pouches. We make bags of markin cloth for containing treated coir dust and check its absorption rate in crude oil.

Weight of the coir dust bag we taken is 45.0861 g. In a beaker we prepare mixture of crude oil and water and mixed it by stirring. Then add the bag to it for 15-30 minute. After absorption, we take out the bag and weighed it.

Weight Gained by the coir dust bag is 107.9720 g.

Weight gain % is 70.54 approx.



4.10 Testing of coir dust handmade paper (round) for absorption in crude oil:

The machine we used for making coir pith and coir-cotton paper is beating machine, sheet form machine and screw press machine. Snaps of machines are given below.

Also the absorption in water & crude oil was shown below.



Beating Machine

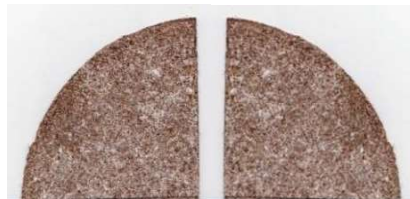


Sheet form Machine

Figure 14 – Beating Machine, Sheet form Machine, Screw press machine,



Screw Press Machine



Handmade coir pith paper

Handmade Coir Pith Paper

Table: 16 Absorption of water and crude oil by handmade paper.

<i>Coir-Cotton Paper/ Absorption</i>	<i>Weight Gain % by paper after Water Absorption</i>		<i>Weight Gain % by paper after Crude oil Absorption</i>	
	<i>20% cotton mixed coir paper</i>	<i>10% cotton mixed coir paper</i>	<i>20% cotton mixed coir paper</i>	<i>10% cotton mixed coir paper</i>
Untreated coir-cotton paper	465.30	494.93	389.86	458.71
Heat treated coir-cotton paper	384.78	371.10	358.78	434.72

4.11 Testing of coir dust of various treated handmade paper for absorption test in crude oil and water :

The absorption rate of **Oven dried** handmade coir pith paper, **Hot plate press** handmade coir pith paper, **Oil treats** handmade coir pith paper, **Latea treats** handmade coir pith papers are checked for both crude oil and water. The weight gain% of each paper after absorption was shown in below:

Table: 17. Variously treated coir dust paper/ sheet exposed to water & C. oil

<i>Treated Coir-Cotton Paper/ Absorption</i>	<i>Oven Dried handmade coir pith paper</i>	<i>Hot Plate press handmade coir pith paper</i>	<i>Oil treated handmade coir pith paper</i>	<i>Latea treated handmade coir pith paper</i>
<i>Weight Gain % by paper after Water Absorption</i>	487.84	446.30	140.20	343.17
<i>Weight Gain % by paper after Crude oil Absorption</i>	370.90	384.16	86.06	266.80

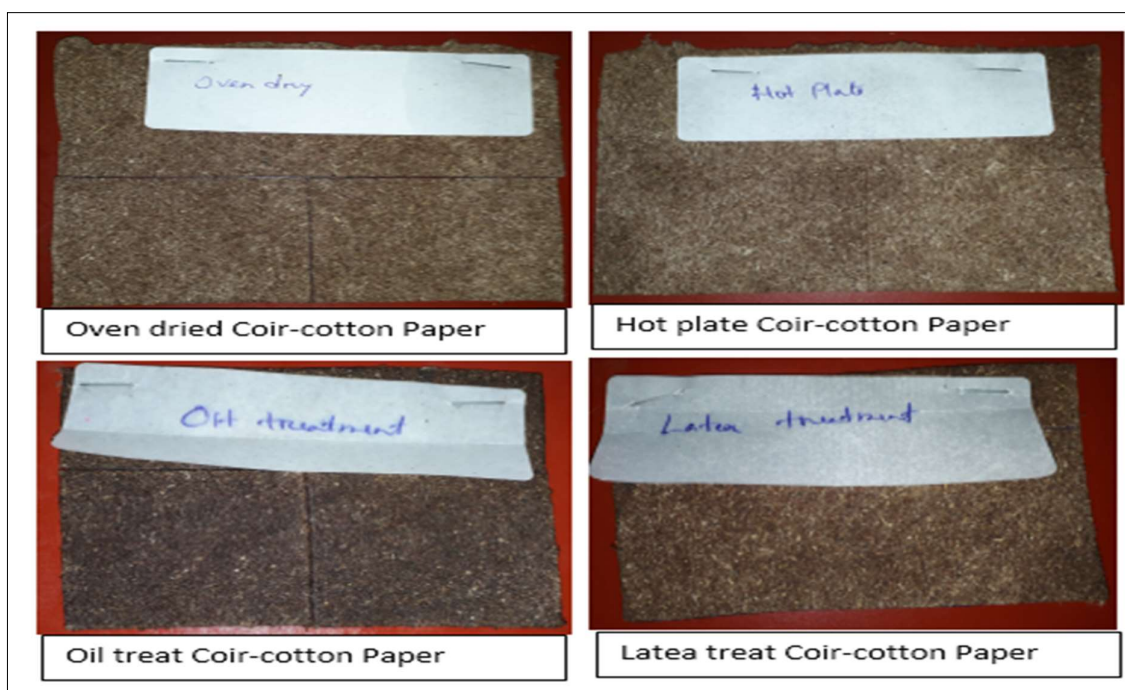


Figure 15- Various treated coir-cotton Paper

4.12 Evaluation of their Physico-chemical properties of Coir Dust, Jute Waste, Cotton Waste :

Physico-chemical properties of CD, JW and CW are represented in Table18. The thickness of cotton waste and jute waste were measured as 0.32 and 1.43 mm and the densities of coir dust, cotton waste and jute waste recorded 0.759 ± 0.2 , 1.55 ± 0.5 and 1.45 ± 0.5 g/mm³. The cellulose content of coir dust, cotton and jute waste were determined and found 35.50 ± 2.0 , 94.50 ± 3.5 and 60.23 ± 4.0 % while lignin content recorded 44.20 ± 3.0 , 15.4 ± 2.5 and 13.3 ± 2 % in cotton and jute waste respectively.

Table 18: Physico- chemical properties of coir dust, cotton and jute waste

Sample	Thickness (mm)	Moisture Content (%)	Ash Content (%)	pH	Density (g/cm ³)	Lignin (%)	Cellulose (%)
Coir dust	-	80.00 ± 5.0	3.40 ± 0.1	5.30 ± 0.5	0.759 ± 0.2	44.20 ± 3.0	35.50 ± 2.0
Cotton waste	0.32 ± 0.05	7.50 ± 0.5	1.75 ± 0.05	7.25 ± 0.6	1.55 ± 0.5	15.4 ± 2.5	94.50 ± 3.5
Jute waste	1.43 ± 0.5	9.93 ± 0.8	0.68 ± 0.05	6.74 ± 0.5	1.45 ± 0.5	13.3 ± 2	60.23 ± 4.0

4.13 FTIR studies of CD, JW, CW and their Composite Mat (OAM):

The representative FTIR spectra of natural waste fibres, coir dust and composite mat (OAM) were shown in Fig. 16. The FTIR spectra of CW fibre and JW fibre [Fig. 16 (a) & (b)] showed a broad and intense band at $\sim 3420\text{ cm}^{-1}$ due to the hydrogen bonded O-H stretching vibration from the cellulose. The IR band at $\sim 2931\text{ cm}^{-1}$ for JW fibre is assigned to $-\text{CH}_2$ anti-symmetric stretching vibration in cellulose and degraded hemicelluloses and lignin. This band is absent for cotton fibre, which may be due to absence of residual lignin resulting in decrease carbon atoms attached to carbon or hydrogen ($-\text{C}-\text{C}-$ or $-\text{C}-\text{H}$) [26]. Band at $\sim 1630\text{ cm}^{-1}$ in both CW and JW fibre is assigned for bending form of absorbed water and due to some involvement of carboxylate group. The $1050\text{--}1250\text{ cm}^{-1}$ region bands involve the C-O stretching vibrations of aliphatic primary and secondary alcohols in cellulose. Both the fibres show a peak at $\sim 889\text{ cm}^{-1}$ due to β -glycosidic linkage of glucose ring of cellulose indicating the typical structure of cellulose [27, 28]. FT-IR spectra of coir dust show characteristics peaks for $-\text{C}=\text{O}$ stretching of the carbonyl and acetyl group in the 4-O-methyl-glucuronoacetyl xylan component of hemicelluloses in coir fibre $\sim 1702\text{ cm}^{-1}$. The band at $\sim 1521\text{ cm}^{-1}$ for coir dust is due to presence of aromatic rings of lignin. A band at $\sim 1247\text{ cm}^{-1}$ was observed for untreated coir fibre which may be attributed to $-\text{C}-\text{O}-\text{C}-$ bond in the cellulosic chain [28]. In the composite board, the individual characteristics FTIR peak of cotton/jute and coir appears with slight change in position and intensity indicating cross linking of coir and natural fibres.

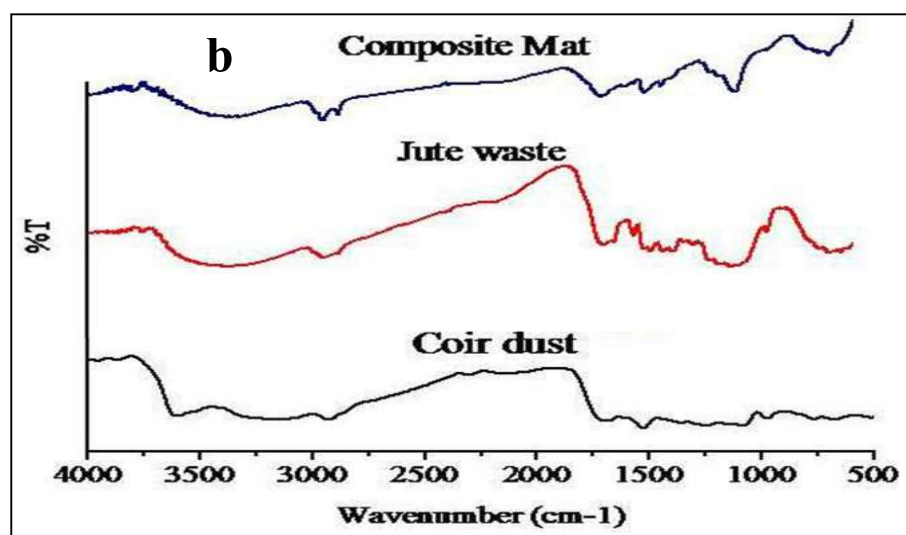
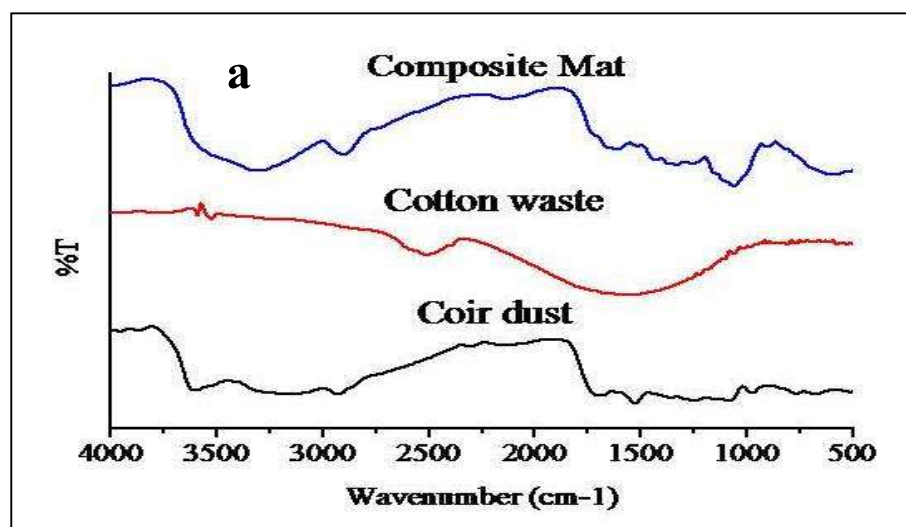


Figure: 16 FT-IR spectra of (a) Cotton waste, coir dust and CD+CW composite mat, (b) Jute waste, coir dust and CD+JW composite mat.

4.14 XRD studies of CD, JW, CW and their Composite Mat (OAM):

The XRD studies of natural waste fibres, coir dust and composite mat (OAM) were carried out to investigate the crystallinity of the samples at different stages and were shown in Fig. 17 (a) & (b). The XRD patterns of CW fibre and JW fibre [Fig. 17 (a) and (b)] showed two peaks representing the planes 101 and 002 at angles 2θ which are subtended at 12.8° and 19.8° respectively, characteristic of cellulose crystalline phase of the fibre [27, 28]. Crystallinity Index (CI) was calculated and it

is found to be 45 % and 58 % for waste JW fibre and CW fibre respectively. This higher value of crystallinity index for CW fibre is consistent with higher value of cellulose with insignificant quantity of lignin. In the XRD of coir dust, an extensive broadening peak with 2θ value in the range of 15.6° and 26.8° are observed due to the characteristic diffraction peak of 101 and 002 planes [29, 30].

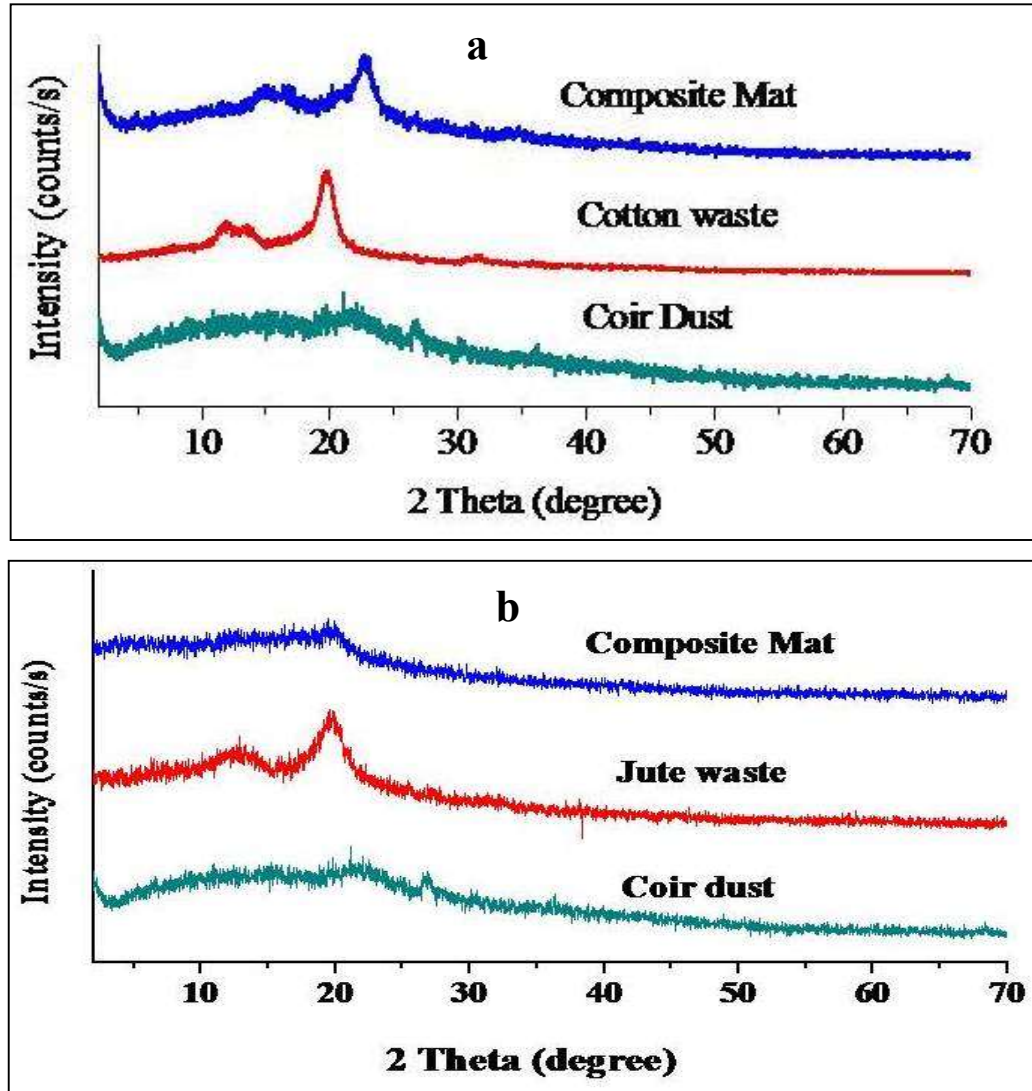


Figure 17: X-ray diffraction spectra of (a) Cotton waste, coir dust and CD+CW composite mat, (b) Jute waste, coir dust and CD+JW composite mat.

The XRD of OAM shows slightly boarder and weaker reflection compared to those of coir and waste fibres which may be attributed to the significant structural modification in the OAM. This modification in comparative intensity and location of reflection peak indicates a three dimensional linkage-formation between coir dust and natural fibre during composite formation.

4.15 TGA studies of CD, JW, CW and their Composite Mat (OAM):

The TGA curves of natural waste fibres, coir dust and composite mat (OAM) were represent in **Fig. 18 (a) & (b)**. The different weight loss percentage obtained from thermogravimetry analysis for particular samples are represented in **Table 18**. The weight loss in the samples occurred in three stages: the first one in the range of 20-120 °C, the second one 120-400 °C and third one 400-650 °C. For the waste fibres, weight loss in the range of 20-120 °C are attributed to the evaporation of absorbed and crystal water molecule associated with the cellulose fibre [31]. The actual degradation occurred in the range of 120-400 °C are assigned for disintegration of polymeric materials such as hemicelluloses, α -cellulose, lignin etc. and the third stage (400-650 °C) is attributed to the carbonization of these polymeric materials [32]. Similarly for coir dust, the first one (7.90 %) refer to the loss of water molecules along with some volatile oily components present in the coir fibre (20-120 °C); second (49.04 %), because of the thermal degradations of polymeric materials (120-400 °C); and the third stage (10.85 %) in the range of 400-650 °C is attributed to carbonization of these polymeric materials [33-35]. It is observed that OAM exhibited weight loss of 56.90 % and 66.49 % for JW mat and CW mat respectively in the transition temperature 120-400 °C compared to 57.81 % and 80.29 % weight loss for jute fibre and cotton fibre individually. Similarly in the transition temperature 400-650 °C, JW composite and CW

composite exhibit weight loss 10.41 % and 10.23 % respectively compared to individual fibres, which was in the range 10.85-19.60 %. This shows that the cellulose of waste fibres and cellulosic coir dust interact strongly forming a three dimensional network and thereby increased the thermal stability of the respective OAM.

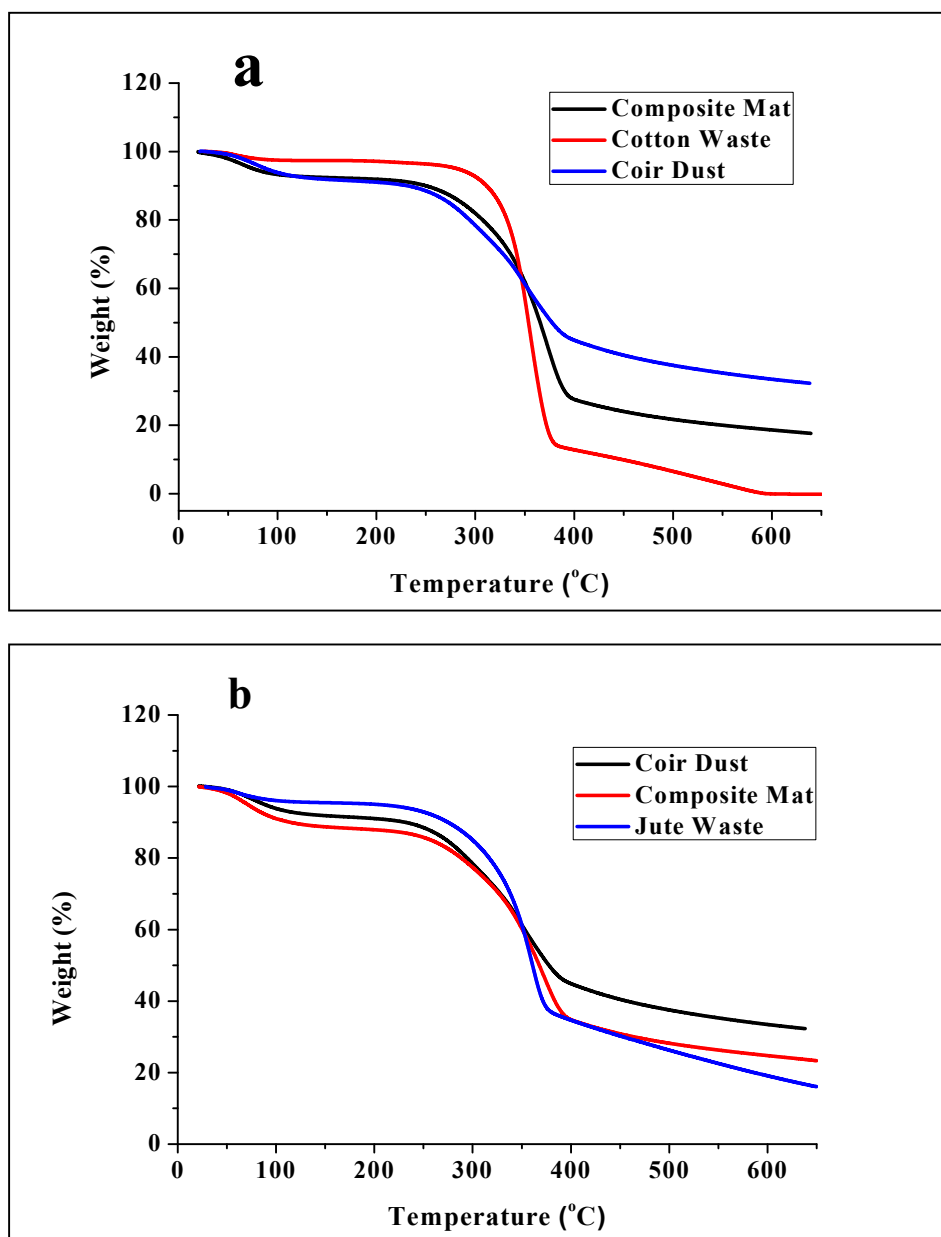


Figure 18 : TGA spectra of (a) Cotton waste, coir dust and CD+CW composite mat, (b) Jute waste, coir dust and CD+JW composite mat.

Table 18: % Weight loss of different samples in various stages

Samples	% weight loss		
	20-120 °C	120-400 °C	400-650 °C
<i>Coir Dust</i>	7.90	49.04	10.85
<i>Jute waste</i>	7.41	57.81	19.60
<i>Cotton waste</i>	5.60	80.29	11.56
<i>Composite Board (CD +JW)</i>	8.98	56.90	10.41
<i>Composite Board (CD +CW)</i>	6.23	66.49	10.23

4.16 Microstructural characteristics (SEM) of CD, JW, CW and their composite

OAM:

Fig. 19 (a-e) & 20 (a-e) represent the SEM and invert SEM of CD, CW, JW and OAM. The SEM of CD [Fig. 19 a (i) & (ii) and 20 a (i) & (ii)] shows that fibre surface was covered with oils, waxes and extractives layer which is the part of natural constituent of fibres(Das et al. 2014). It also observed from the SEM of coir dust that very rough surface and shallow pits were also visible of coir dust [36]. Fig. 19 (b) & 20 (b) represented the SEM of cotton waste fibre and observed that the fibres are long, continuous, thin and whitish in colour. Also observed that, the fibres are forming a tough network without adding of any binder as well as no pores or longitudinal cracks are perceptible on the surface. Fig. 19 (c) & 20 (c) represents the SEM of JW fibre. Jute fibres are continuous, uniform and cylindrical in shape

which is comparable with CW fibre. Some bundle forms of fibre are also observed [37]. The SEM of the composite mat prepared from cotton waste and coir dust are presented in Fig. 19 (d) & 20 (d). A strong network of cotton and coir fibres consistently bonded by guar gum solution has been seen. This similar network of two natural fibres may be due to superior fibrillation taken place during stock preparation in beating. Fig. 19 (e) & 20 (e) shows the SEM of composite mat made from coir dust and JW. In jute fibres, comparatively larger in diameter, fibres are clearly visible. The coir fibres are fixed with the jute fibres uniformly forming a strong network of jute and coir fibres. During stock preparation, the void spaces between coir and jute fibre are occupied by guar gum. Due to some void spaces between coir and natural fibres, oil absorbency may also increased. The gum solution used in beating operation during stock preparation helps to enhance the cohesiveness of both the fibres. Although, natural fibres have similar morphology, but there are differences among them which may be because of the deviation of morphological properties like number of fibre cells, cell wall size, size and shape of the lumen etc. Therefore, different fibre plants show different characteristics as well as mechanical properties [38]. Figure 21 (a-f) represent the surface plot diagrams of coir dust, cotton waste, jute waste and composite mat made from mixture of coir dust and cotton waste or jute waste, which reflect their surface morphologies. Some rough stimulus were seen randomly throughout in the micrographs of coir dust, cotton waste and jute waste, [Fig. 21 (a), (b), (c) & (d)], whereas they are low distinguishable as well as with relatively smoother surface in the micrographs of absorbent mat which is made from the mixture of CD and CW or JW [Fig. 21 (e) & (f)].

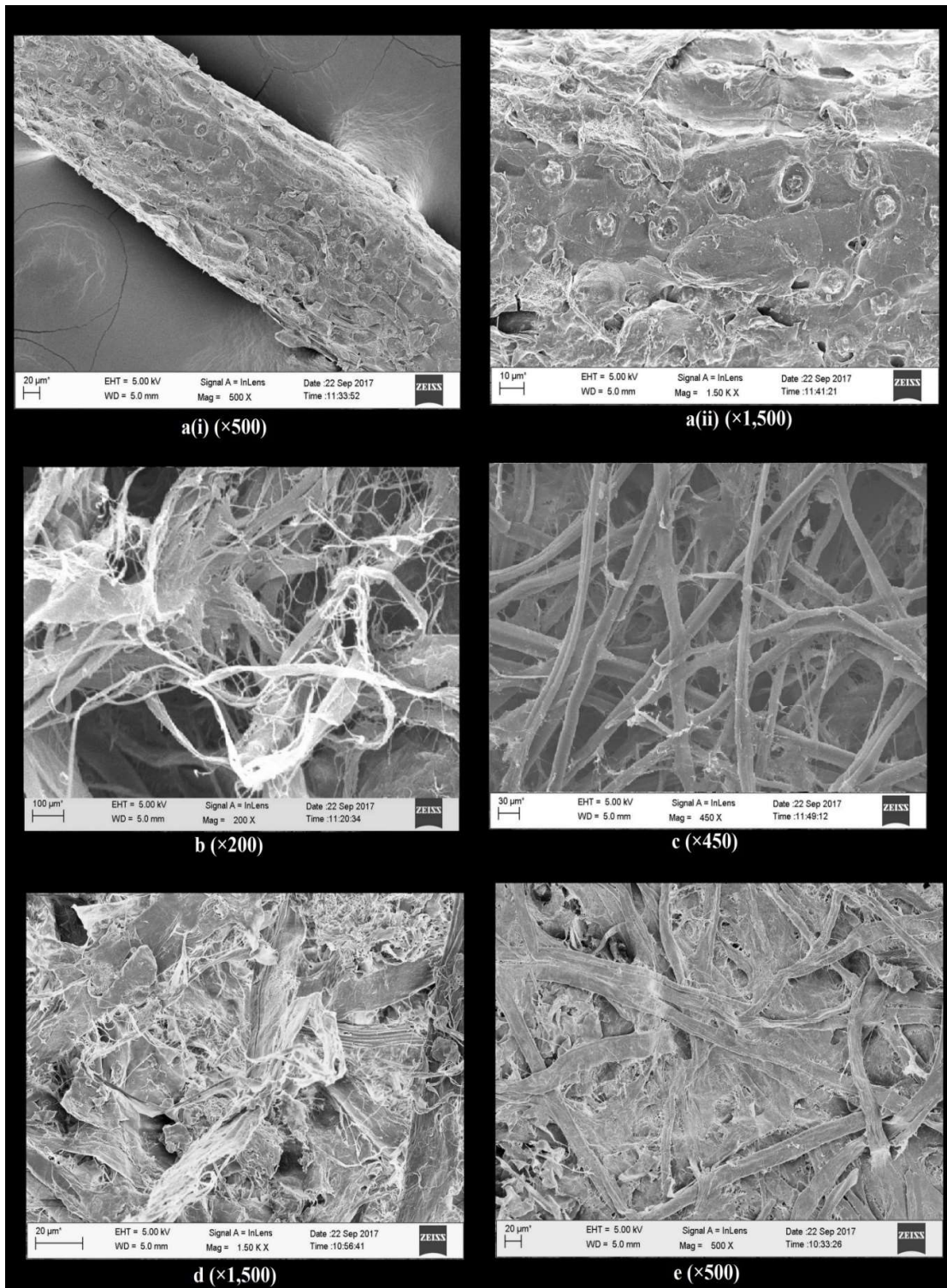


Figure 19: SEM of (a) Coir Dust, (b) Cotton waste fibre, (c) Jute waste fibre, (d) Composite made from Coir dust + cotton waste, (e) Composite made from Coir dust + jute waste.

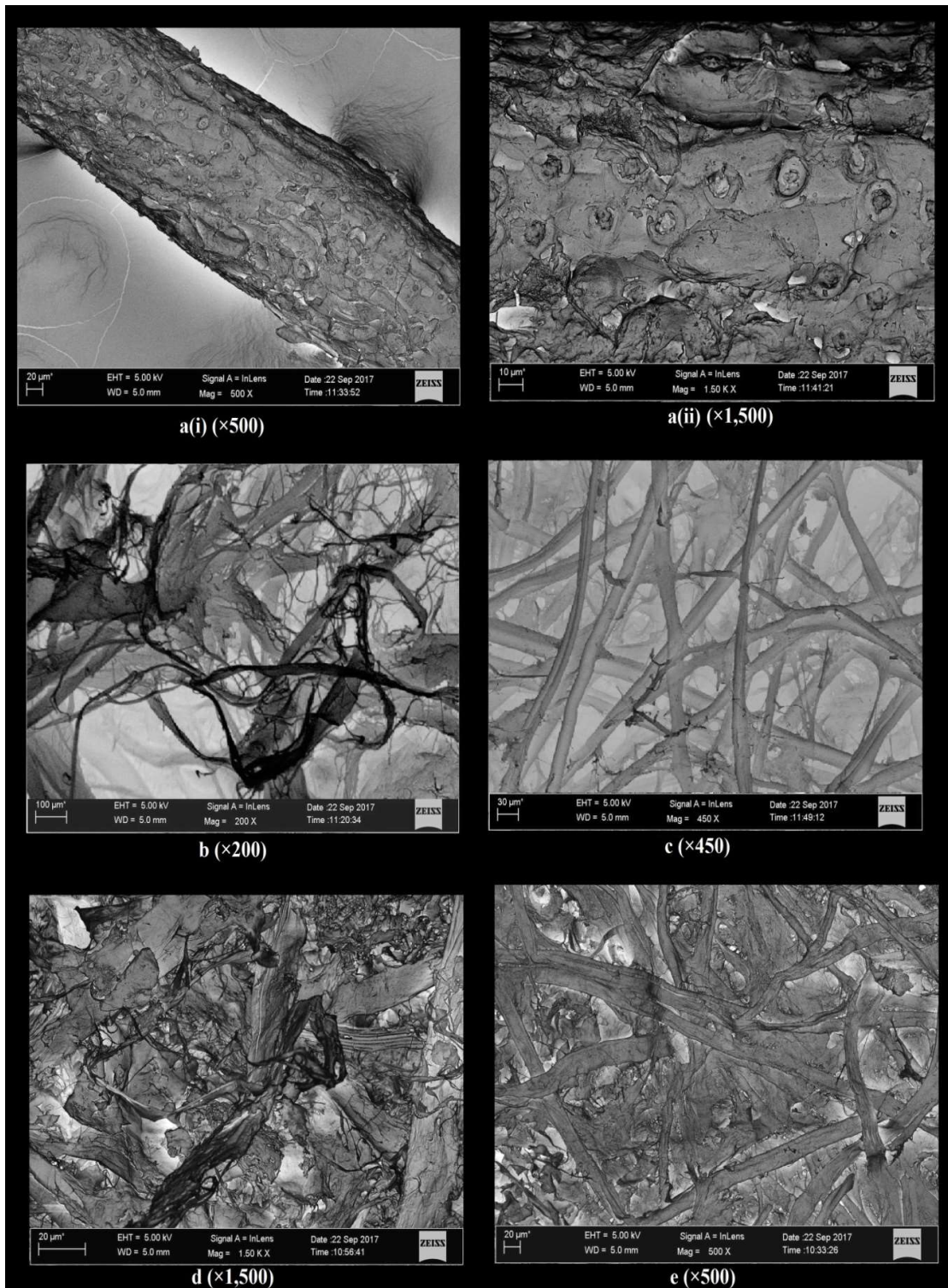


Figure 20: Invert SEM of (a) Coir Dust, (b) Cotton waste fibre, (c) Jute waste fibre, (d) Composite made from CD + CW, (e) Composite made from CD+JW.

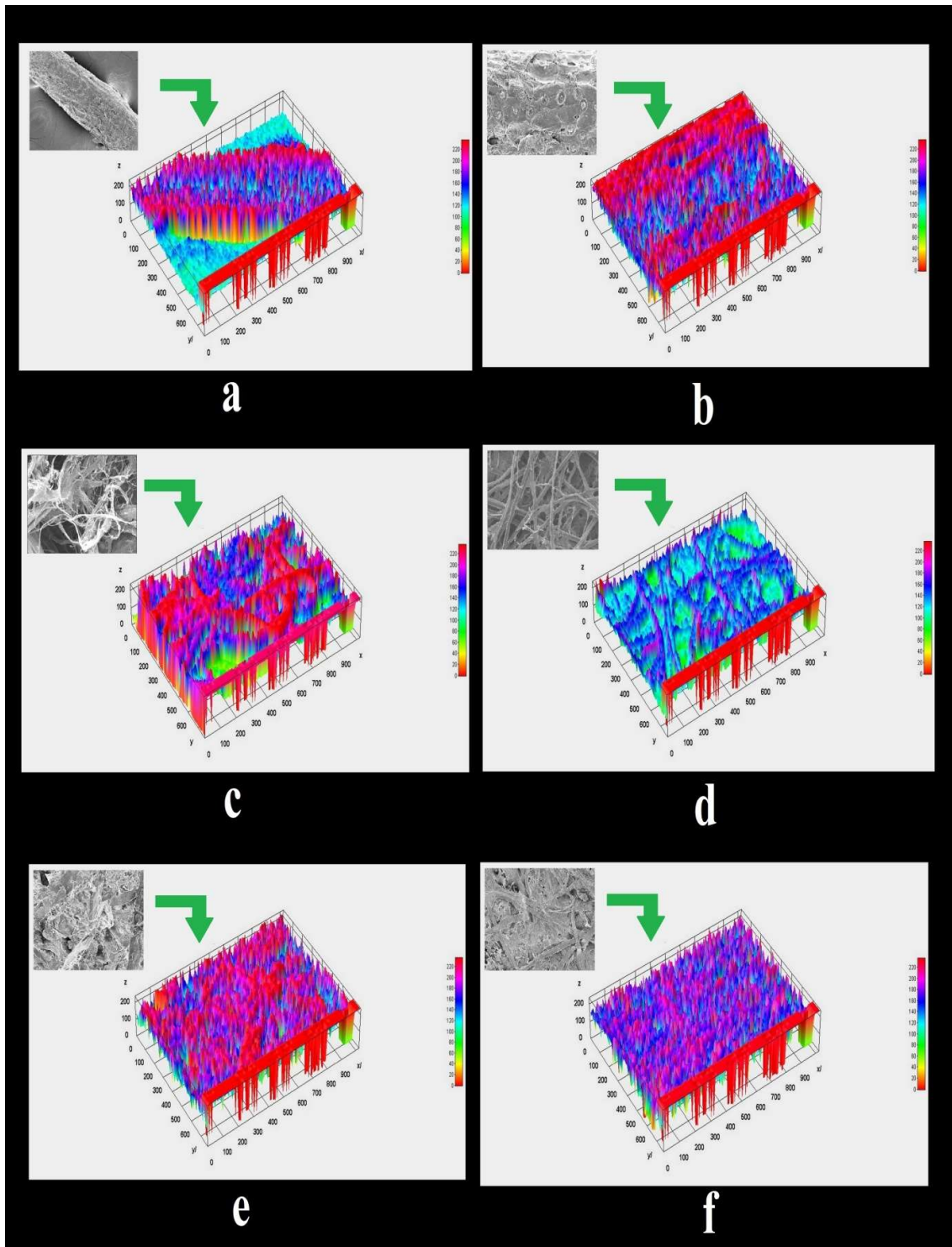


Figure 21: Surface SEM analysis of (a) Coir Dust, (b) Cotton waste fibre, (c) Jute waste fibre, (d) Composite made from CD + CW, (e) Composite made from CD+JW.

4.17 Mechanical Properties of JW, CW and their Composite OAM:

The mechanical properties like ultimate tensile strength (UTS), elongation of CW and JW are represented in **Table 19**. The UTS of JW and CW were recorded as 250.62 ± 6.0 and 235.00 ± 5.0 MPa respectively which are shown in the **Fig. 22**. The superior tensile strength may attribute to presence of cellulose substance in the natural fibre [37]. Rowell studied that lower cellulose content don't contribute much towards the tensile strength [39]. Likewise, similar elongation values were also recorded for jute waste and cotton waste which may be stated as 12.35 ± 2.5 and 11.65 ± 2.0 % respectively. The mechanical or physical strength properties of natural fibre materials were found with similar results without showing much deviation which is compatible for making composite mats.

Table 20 represents the mechanical properties of the OAM prepared from the mixture of coir dust and cotton waste or jute waste. Similar densities were recorded which 0.258 ± 0.03 and 0.249 ± 0.06 g /cm³ for the mats are made from the mixture of CD & CW and CD & JW respectively in the ratio of 85:15. Different densities were recorded in mats with differing ratios i.e. 90:10, 85:15 and 80:20 may be due to the gradual increment of natural fibre [37]. The ultimate tensile strength properties of OAM prepared in combination of CD and CW or JW at different stock ratios have also been shown in **Table 20**. It has been observed in the **Fig. 23**, the OAM manufactured from CD & CW showed comparatively lower tensile strength (43.86 ± 3.5 MPa) compare to that of mats made from CD & JW (49.17 ± 4.0 MPa). Natural fibre improved the mechanical strength properties, which is due to the uniform bonding between natural fibre and coir dust fibre [37]. Natural fibre forms a regular network with the coir fibre, that causes improved ultimate strength properties. Senthil also observed similar results along with increased tensile strength of the composite mats due to the presence of cellulose content in the natural fibre [40]. Rowell also observed the same result in the composite mats

prepared from the mixture of CD & CW/ JW [39]. A natural binder i.e. guar gum was used in the mixture sock, which integrates to improve the physical strength of the final absorbent mat product. **Table 20** represents certain mechanical properties like elongation, folding and bursting properties of the absorbent mats, where 85:15 ratio was found to be encouraging with superior results compared to other ratios. Hence, the above ratio of coir dusts and natural fibres has been found to be optimum to get high mechanical strength properties in the absorbent mats.

Table 19: Mechanical properties of jute and cotton waste fibre

<i>Sample</i>	<i>UTS (MPa)</i>	<i>Elongation (%)</i>	<i>Breaking Load in tensile test (N)</i>
Jute waste	250.62±6.0	12.35±2.5	3215±30
Cotton waste	235.00±5.0	11.65±2.0	2955±25

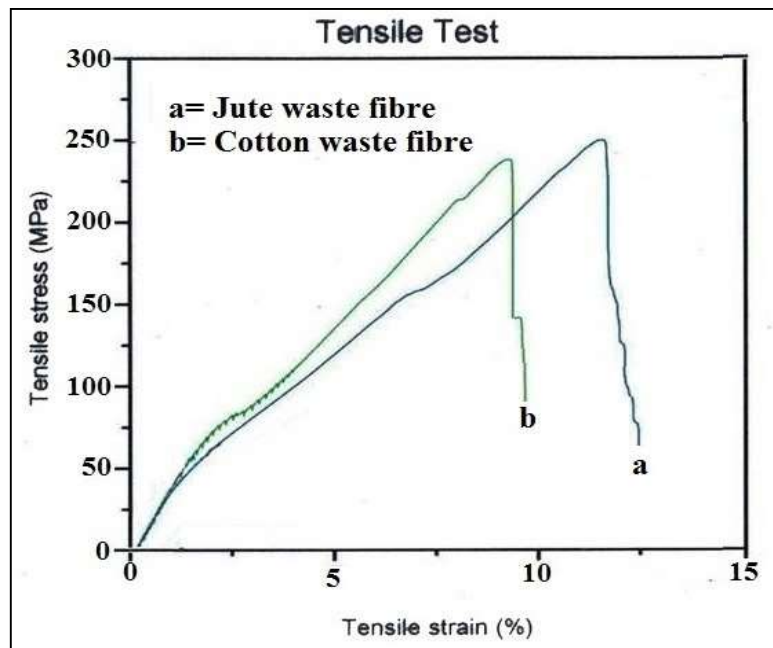


Figure 22: Tensile strength of jute waste fibre and cotton waste fibre.

Table 20: Mechanical properties of composite made from coir dust, cotton and jute waste:

Sample	Moisture content (%)	Density (g/cm³)	UTS (MPa)	Elongation (%)	Breaking Load in tensile test (N)	Folding endurance (DF/min)	Bursting strength (kg/cm²)
CD+ CW (90:10)	39.40±1.2	0.244±0.02	30.76±3.0	4.78±0.5	2340±15.0	440±5.0	5.60±1.5
CD+ CW (85:15)	36.10±1.0	0.258±0.03	43.86±3.5	18.15±1.5	2525±25.0	1446±15.0	8.80±2.0
CD+ CW (80:20)	32.80±0.8	0.281±0.02	29.42±3.0	4.35±0.8	2450±10.0	406±9.0	6.80±1.8
CD+ JW (90:10)	45.20±1.3	0.238±0.05	35.85±3.5	4.00±1.0	1940±10.0	98±4.0	3.30±0.5
CD+ JW (85:15)	42.00±1.2	0.249±0.06	49.17±4.0	23.86±2.0	2895±15.0	1850±20.0	9.70±1.0
CD+ JW (80:20)	38.20±1.1	0.258±0.05	38.38±3.0	17.08±1.5	2950±15.0	750±5.0	6.20±1.2

Coir Dust=CD, Cotton waste =CW, Jute waste =JW

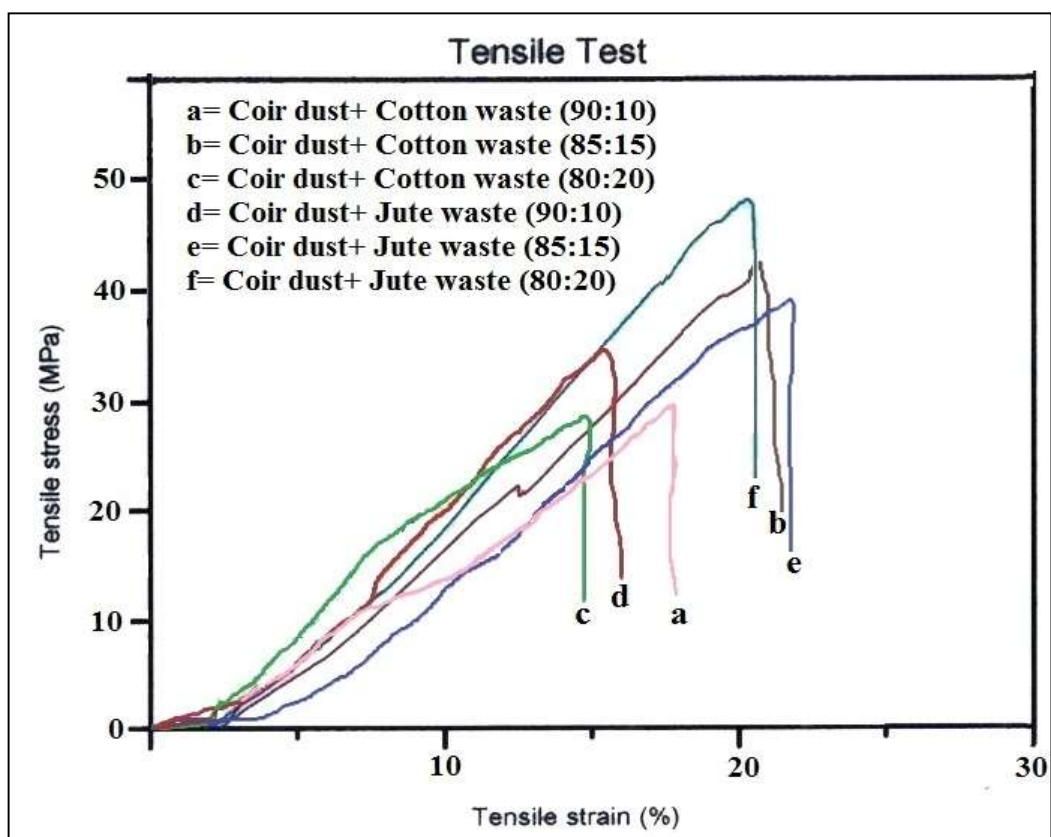


Figure 23: Tensile strength of composite made from Coir dust and Cotton waste (90:10).

a) Coir dust and Cotton waste (90:10), b) Coir dust and Cotton waste (85:15), c) Coir dust and Cotton waste (80:10), d) Coir dust and Jute waste (90:10), e) Coir dust and Jute waste (85:15), f) Coir dust and Jute waste (80:10).

4.18 Oil Absorption Properties of OAM:

Table 21 represents the oil absorption properties of the OAM prepared from the mixture of CD and CW or JW in different oils like crude oil, diesel oil, hydraulic oil, transformer oil, Mobil oil. Oil absorption values also show comparable & superior results in the mats made from the CD & CW and CD & JW in the ratio of 85:15 at 20-30 °C. Presence of natural fibre as well as more cellulose containing cotton fibre such as CD & CW composite mat may show better oil absorbent results than the composite made from CD & JW. It is evident that oil absorbency is better in low density oil than the high density ones.

Table 22 represents the comparison of various commercial or known oil absorbents model with OAM. Oil absorbency of OAM shows better performance than commercial or known oil absorbents model in different types of oil.

Table 21: Oil Absorption properties of composite made from coir dust, cotton waste and jute waste

Sample	Crude Oil (g/g)	Diesel Oil (g/g)	Hydraulic Oil (g/g)	Transformer Oil (g/g)	Mobil Oil (g/g)
After 20 min at 20-30 °C					
CD+ CW (90:10)	21.0±2.0	17.1±1.8	21.3±2.1	17.5±1.5	18.0±1.5
CD+ CW (85:15)	21.8±2.0	19.2±2.0	19.0±1.5	18.6±1.8	20.4±2.0
CD+ CW (80:20)	18.1±1.5	14.9±1.0	17.1±1.5	14.3±1.0	15.2±1.5
CD+ JW (90:10)	18.9±1.5	19.6±1.5	18.4±2.0	18.4±1.5	16.6±2.0
CD+ JW (85:15)	19.6±2.0	19.1±1.6	19.6±2.0	18.6±1.5	19.6±1.8
CD+ JW (80:20)	18.4±2.5	17.9±2.3	17.5±1.5	15.4±0.5	18.9±1.5

Oil Density:

**Crude Oil=880 kg/m³, Diesel Oil= 820 kg/m³, Hydraulic Oil= 874 kg/m³,
Transformer Oil= 800 kg/m³, Mobil Oil= 840 kg/m³**

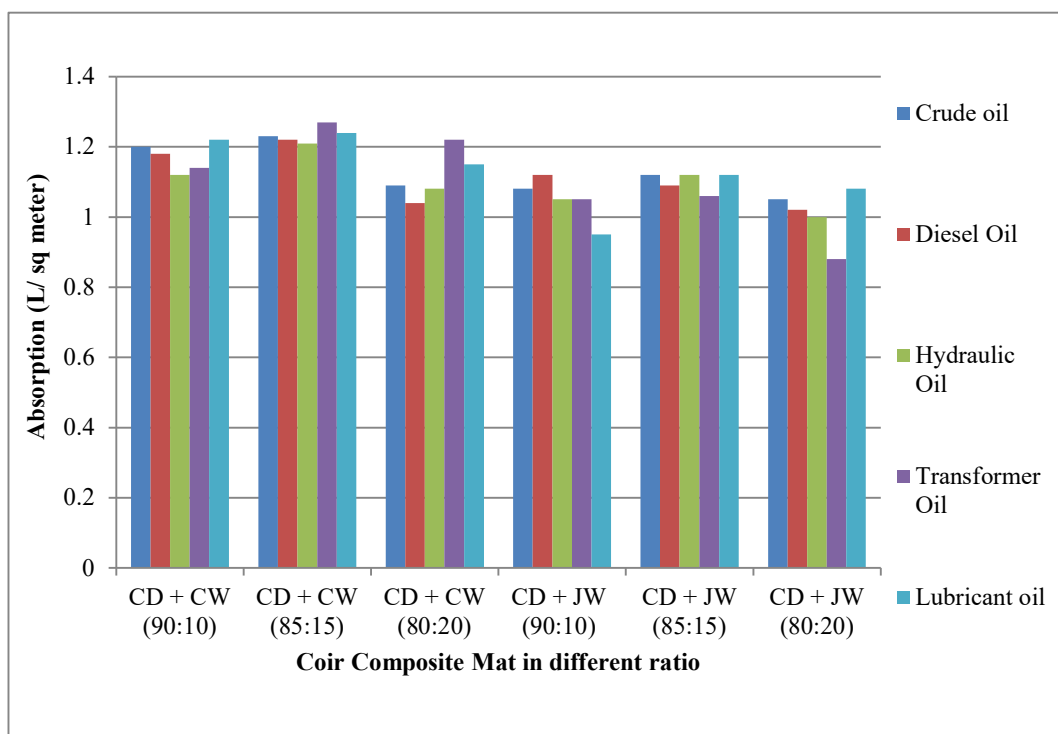


Figure 24: Absorption of various tested oil by Oil Absorbent Mat (OAM)

Table 22: Comparison of Various Oil Absorbents

Oil Absorbent Media	Oil	Oil Absorbency (g/g)	Reference
Oil absorbent mat (OAM) [CD+ CW (85:15)]	Crude	21.8±2.0	Present work
	Diesel	19.2±2.0	
	Hydraulic	19.0±1.5	
	Transformer	18.6±1.8	
	Mobil	20.4±2.0	
Swellable Porous PDMS	Diesel	12.0	41
	Crude Oil	9.0	
PDMS sponge	Transformer	4.3	42
Cotton towel	Diesel	5.0	43
Commercial PP nonwoven mat	Hydraulic	11.7	44
	Crude	8.1	

Polyvinyl-Alcohol Formaldehyde Sponges	Hydraulic	12.8	44
	Crude	12.9	

4.19 Recovery and Reusability:

Recovery and reusability is a significant factor for oil absorbent. In order to measure the possible for oil recovery from OAM, we used mechanical extraction approach. With the help of hydraulic press at 25 N of pressure, OAM were compressed while suspended over, until no further oil extruded from the compressed mat. The resultant oil was weighed immediately, and this process was repeated for 10 cycles. Interestingly, the absorption capacity of OAM started higher (19.8 ± 0.1 g/ g) and then capacity decreased (11.6 ± 0.2 g/g) slowly after 10 cycles. **Fig.25** represents the recycle oil absorption capacity of OAM (CD & CW; 85:15) in each cycle. After 10 cycles, the absorption capacity of OAM was retained at 60 % compared to the first cycle. But after 4 to 5 cycle cracks are observed on OAM and it goes on deterioration.

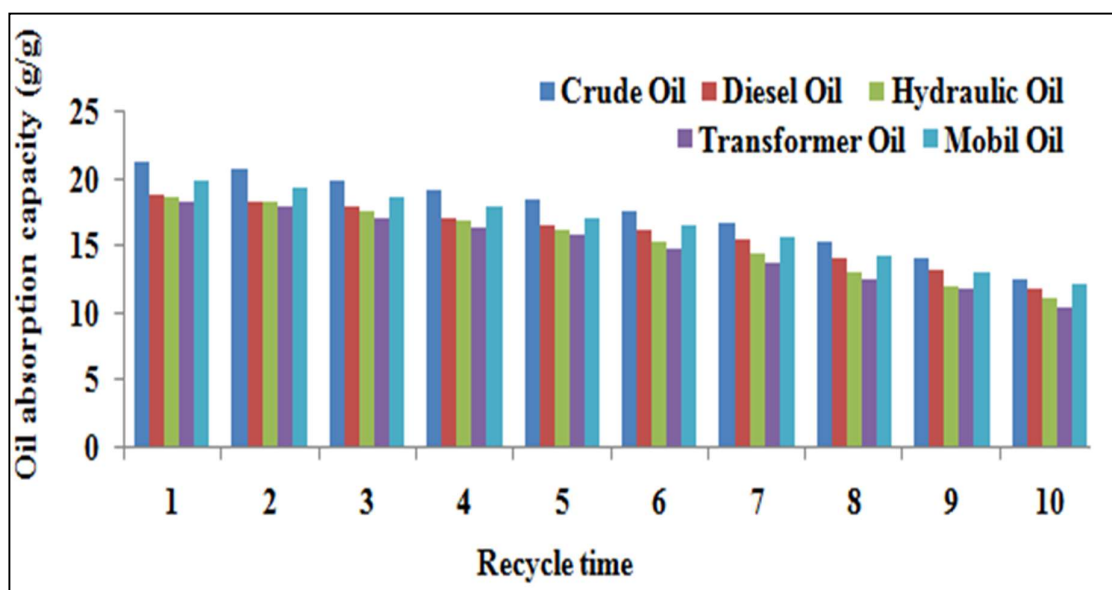


Fig. 25: Recycle oil absorption capacity of composite made from coir dust and cotton waste (85:15).

Chapter 6

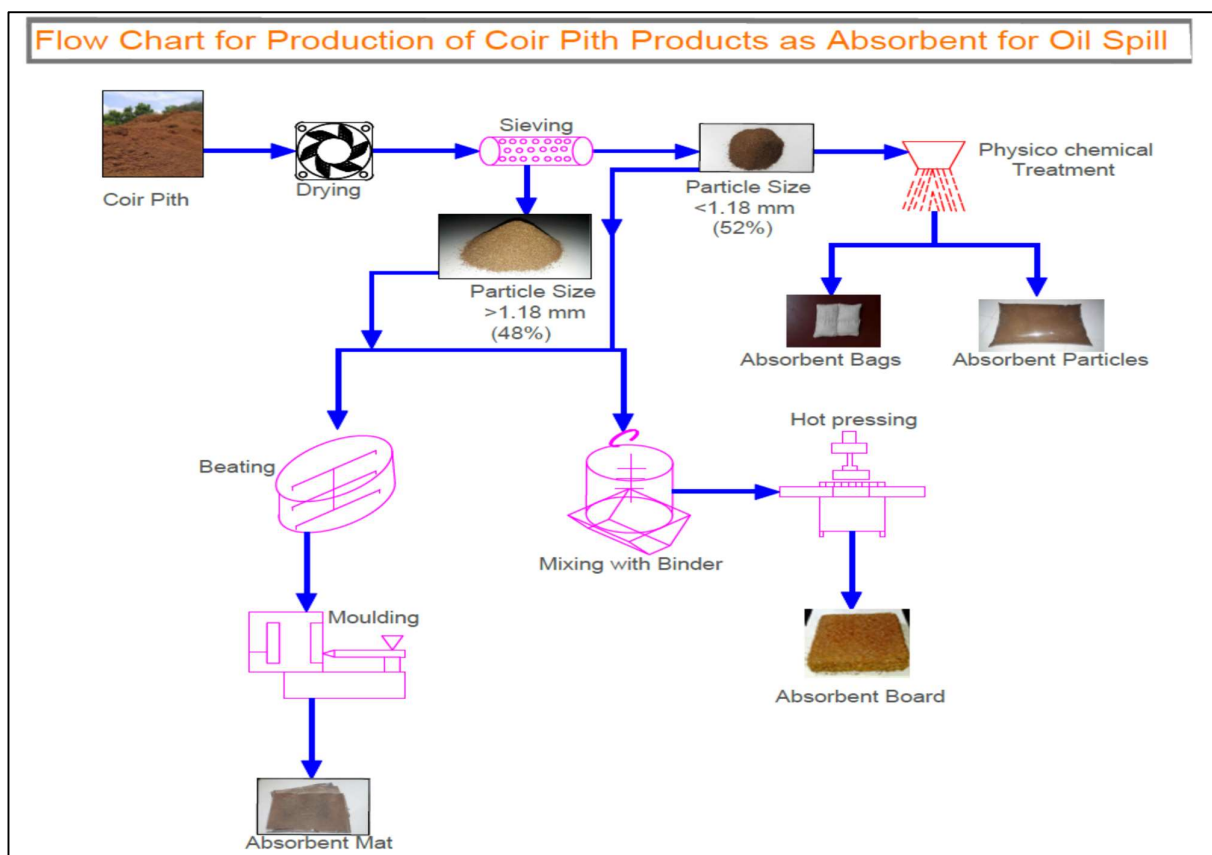
Conclusion

Natural organic sorbents, such as coir dust, are environmental-friendly, biodegradable and inexpensive materials if compared to polymer synthetic materials. Some of the natural organic sorbents have good sorption capacities, but they also sorb water as well (low hydrophobicity). This is a disadvantage when used in water environments. Hydrophobicity of the sorbent is an important parameter that determines the effectiveness of sorbent. Some physicochemical treatments were done to improve the hydrophobicity of coir dust such as heating, acetylation, alkali treatment or Acylation (Esterification), while in esterification, 30% Coir-oleate can serve as an alternative for oil spill due to its easy modification, abundance and biodegradability with more oleophilic character as compared to other.

From the present study it may be concluded that coir dust, a waste produced from the coir industry, can be converted to the form of pouches/bag, block, mat/pad by addition of natural fibre and also by adopting certain mechano-chemical progression. The mats or sheets prepared from blending of cotton wastes or jute wastes with coir dusts exhibited better oil absorbency, with adequate physical strength properties, and that makes them suitable to be used in case of oil spills in oil exploration, industries, transportation and in machine/vehicle maintenance works etc. As the mat/pad is biodegradable or recyclable in nature, it can be easily disposed to soil or environment after extracting the oil from the mat/pad. Recycling of such industrial wastes for making useful products not only helps the society, but also helps to conserve the ecosystem and environment.

COIR PITH PRODUCTS as ABSORBENT

Flow Chart for production of Coir pith Products as Absorbent for oil spill



Photographs of Developed Products preparation for field trial:



Figure 26: Developed products from coir dust

Patent Filed in India (Patent Application No. 0015NF2019):

A patent entitled “High Oil Absorbing Mat/ Pad” was filing jointly by CSIR NEIST and Coir Board.

Authors:

JAYANTA JYOTI BORA (CSIR-NEIST),
PALLAV SAIKIA (CSIR-NEIST),
TRIDIP GOSWAMI (CSIR-NEIST),
DIPANKAR NEOG (CSIR-NEIST),
ANITA DAS RAVIDRANATH (COIR BOARD),
SUBODH CHANDRA KALITA (CSIR-NEIST),
PINAKI SENGUPTA (CSIR-NEIST).

Poster Presentations:

1. A poster was Presented in the **Materials Research Society of India (MRSI)**, North-East Chapter conference on The Frontier Chemical Biology, 26-28 June, 2018 jointly organised by CSIR-NEIST and Assam Science Society Jorhat branch. The Poster was presented on 26th June, 2018 at CSIR-NEIST. Title of the poster was **“Characterisation and Physico-chemical Treatment of Coir Pith to Improve its Hydrophobic and Oleophilic Character for Sorption of Oil Spillage”**.

Authored by Bishnu Sahu, Dipankar Neog, Lakshi Saikia, Dhanjit Das and Jayanta Jyoti Bora

2. Another Poster was presented on 21st February, 2020 in the **International Conference on Engineering Sciences & Technologies for Environmental Care (ESTEC-2020), February 20-22, 2020** organised by CSIR-NEIST, JORHAT. Title of the Project was **“Flow Behaviours Through the Physico-Chemically Modified Coir Dust Based Mat for Application of Prevention of Oil Spillage”**.

Authored by Bishnu Sahu, Dipankar Neog, Dhanjit Das and Jayanta Jyoti Bora.

The poster presentations were shown below:



Characterisation and Physico-chemical Treatment of Coir Pith to Improve Its Hydrophobic and Oleophilic Character for Sorption of Oil Spillage



Bishnu Sahu¹, Dipankar Neog¹, Lakshi Saikia², Dhanjit Das¹ and Jayanta J Bora^{1*}

¹General Engineering Group, Engineering Science & Technology Division; ²Advanced Materials Group, Materials Science & Technology Division
CSIR-North East Institute of Science and Technology, Jorhat – 785006, Assam, India

Introduction

Petroleum is the soul of any modern industrial society. It is the paramount source of energies and raw material for synthetic polymers and chemicals. But during the processing of crude oil exploration to petroleum products, transportation and refining it creates different types of environmental pollution.

For oil spill cleanup, sorbent is used for preventing the pollution or rectifying the tainted land or water. Sorption is the famous technique for cleanup of oil spills. Coir pith, a natural organic sorbent and a waste product generating from the coconut husk and fibres extraction, could be used for cleanup oil spill which is environment-friendly, biodegradable and economical material with compared to the synthetic materials which are used as sorbent.

Objectives & Methods

Study on the characterisation of coir pith such as calorific value, moisture content, ash content, cellulose content, lignin content, pH value, alkali solubility (1 % NaOH solution), cold water solubility, hot water solubility, bulk density, porosity, water holding capacity etc according to standard methods (TAPPI, 1980; AOAC, 1975) shown in Table 1.

Study different Physico-chemical treatments of coir pith as an absorbent to be used for prevention of oil spillage effect with reference to NE oil industries.

For improving hydrophobicity and oleophilic character of coir pith, physico-chemical treatments such as heating at relative low temperature, acid treatment, alkali treatment, acetylation and acylation treatment were done and absorption in Water and oil were tested as per ASTM method. The properties of treated coir pith were characterized by FTIR and SEM.

Results & Discussions

Table 1. Characterization of Coir pith

Assay	% Content in coir pith/ Value	
	Source 1	Source 2
Calorific value	3820 kcal/kg	3825 kcal/kg
Ash content	13.27 %	13.35 %
pH of coir pith in water	6.64	6.65
Hot water solubility	13.9 %	14.3 %
Cold water solubility	10.5 %	11.2 %
Solubility in dil. Alkali (1%)	17 %	17.2 %
% Swelling of coir pith in H ₂ O	65.88 %	65.72 %
Cellulose content	36.36 %	35.9 %
Lignin content	44.9 %	44 %
Bulk density	156.39 kg/m ³	155.20 kg/m ³
Total porosity	74.4 %	75.75 %
Aeration porosity	18.8 %	20.05 %

Table 2. Particle Size Distribution of 1kg of Coir pith

Average particle Size (mm)	Weight of coir dust (gm)	Weight %
+10	30	3.0
-10 to +5.6	72	7.2
-5.6 to +4.75	70	7.0
-4.75 to 2.36	110	11.0
-2.36 to +1.18	196	19.6
-1.18 to +0.600	236	23.6
-0.600 to +0.300	222	22.2
-0.300	64	6.4

Table 3. Absorption of water and crude oil by different treated & raw coir pith

Size of coir dust +0.600mm	Raw Coir pith	Heat treated Coir pith	Acid treated Coir pith	Acetylated Coir pith	Alkali treated Coir pith
Weight absorbed by Coir pith by water absorption (gm/gm of sorbent)	6.9824	6.1521	7.0780	5.4339	5.0994
Weight Absorbed by coir pith by crude oil absorption (gm/gm of sorbent)	6.0310	6.8351	7.6750	7.7777	9.0617

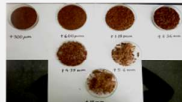


Fig. 1 Particle Size Distribution of Coir pith

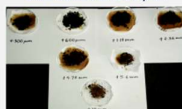
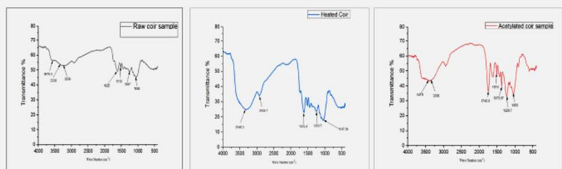
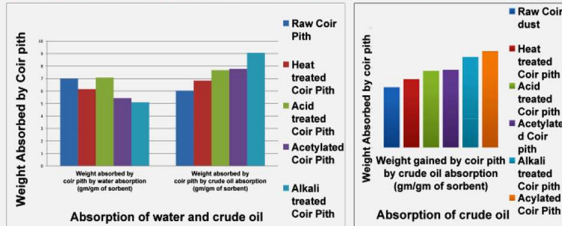


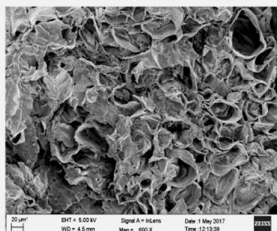
Fig. 2 Crude oil Absorption by coir pith particle

Absorption of Water and Crude oil by raw & treated coir pith particles

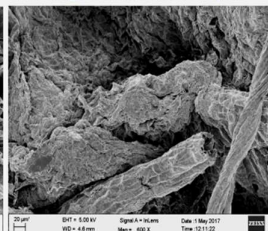


The FT-IR results indicate that coir pith has undergone modification by means of acetylation reaction. The decrease in the intensity of the O-H group absorption bands at 3419cm⁻¹ in spectrum of acetylated coir pith indicated that the acetylation was occurred to the O-H groups. This means hydrophobic character increases in acetylated coir pith.

SEM Image of Acetylated coir pith



SEM Image of heat treated coir pith



- The coir pith sorbent was characterized by standard methods (TAPPI,1980; AOAC,1975) and the results of two sources of coir pith (Source 1 from Biswanath Chari and source 2 from Nalbari, Assam) are shown in Table 1.
- Particle size distribution of coir pith shows that -1.18 mm to +0.300 mm particles are maximum present in coir pith.
- The absorption graph shows that water absorption capacities were lower from 6.98 g-water/g-sorbent to 6.15, 5.43 and 5.09 g-water /g-sorbent for heated coir, acetylated coir and alkali treated coir pith respectively, while crude oil absorption is maximum in acetylated coir pith from 6.03 g-oil/g-sorbent to 9.63 g-oil/g-sorbent, in alkali treated coir pith it is 9.06 g-oil/g-sorbent and in acetylated coir pith it is 7.78 g-oil/g-sorbent.
- Sem image of acetylated coir pith indicates the empty pores which means the absorption capacity increases.

Conclusions

- The hydrophobicity of the Coir pith increases by heating, acetylation, alkalization and acylation, while the absorption of crude oil is maximum in Acetylated coir pith.
- Coir pith was Successfully esterified using oleoyl chloride by the replacement of -OH groups of polymer backbone with that of acyl groups so as to achieve hydrophobic behavior to interact highly with the similarly hydrophobic oil molecules.
- FTIR and SEM characterization were used to investigate the surface morphology and chemical compositions acetylated coir pith. Based on this result, it is undoubtedly true that the modified coir pith could be used for the cleanup of oil spilled in aquatic environments.
- Further studies are need for the design and development of coir pith captr in the shape of membrane/blanket/sheet/pad/block to deal with oil spillage.



Acknowledgement

Authors are thankful to the Director of CSIR-NEIST, Jorhat Assam and also thankful to Coir Board (Min. of MSME) Govt. of India (Project no. CLP-0285) for financial support.

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Figure 27: MRSI poster presentation



FLOW BEHAVIOR THROUGH THE PHYSICO-CHEMICALLY MODIFIED COIR DUST BASED MAT FOR APPLICATION OF PREVENTION OF OIL SPILLAGE

Bishnu Sahu¹, Dipankar Neog¹, Dhanjit Das¹, Jayanta J. Bora¹

¹ General Engineering Group (ESTD), CSIR-NEIST, Jorhat, Assam, India

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INTRODUCTION

Petroleum is the soul of any modern industrial society. It is the paramount source of energies and raw material for synthetic polymers and chemicals. But during the processing of crude oil exploration to petroleum products, transportation and refining it creates different types of environmental pollution.

For oil spill cleanup, sorbent is used for preventing the pollution or rectifying the tainted land or water. Sorption is the famous technique for cleanup of oil spills. Coir pith, a natural organic sorbent and a waste product generating from the coconut husk and fibres extraction, could be used for cleanup of oil spill which is environment friendly, biodegradable and economical material with compared to the synthetic materials which are used as sorbent.

Particles Size distribution of the coir pith, size wise absorption, characterization of coir pith was recorded.

Coir dust/pith was Physico-Chemically Modified to particles, blocks and mats for cleanup of oil spillage and checked the absorption in water, crude oil and other oils.

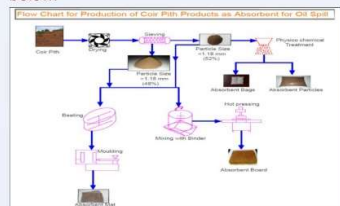
METHODS

Study on the characterisation of coir pith such as calorific value, moisture content, ash content, cellulose content, lignin content, pH value, alkali solubility (1 % NaOH solution), cold water solubility, hot water solubility, bulk density, porosity, water holding capacity, etc according to standard methods (TAPPI, 1980; AOAC, 1975) shown in Table 1.

Permeability of water flow in coir pith particles and mat are studied using Darcy's law.

Study different Physico-chemical treatments of coir pith as an absorbent to be used for prevention of oil spillage effect with reference to NE oil industries.

Flow Chart for Production of coir pith products as Absorbent for oil spill shown below:



RESULTS & DISCUSSION

Table 1. Characterization of Coir pith

Assay	% Content in coir pith/ Value	
	Source 1	Source 2
Calorific value	3820 kcal/kg	3825 kcal/kg
Ash content	13.27 %	13.35 %
pH of coir pith in water	6.64	6.65
Hot water solubility	13.9 %	14.3 %
Cold water solubility	10.5 %	11.2 %
Solubility in dil. Alkali (1%)	17 %	17.2 %
% Swelling of coir pith in H ₂ O	65.88 %	65.72 %
Cellulose content	36.36 %	35.9 %
Lignin content	44.9 %	44 %
Bulk density	156.39 kg/m ³	155.20 kg/m ³
Total porosity	74.4 %	75.75 %
Aeration porosity	18.8 %	20.05 %
Water holding capacity	55.6 %	55.7 %
Permeability of coir pith particles	0.0424 cm/s	0.0343 cm/s
Permeability of coir pith absorbent Mat	0.0161 cm/s	----

Table 2. Particle Size Distribution of 1kg of Coir pith

Average particle Size (mm)	Weight of coir dust (gm)	Weight %
+10	30	3.0
-10 to +5.6	72	7.2
-5.6 to +4.75	70	7.0
-4.75 to 2.36	110	11.0
-2.36 to +1.18	196	19.6
-1.18 to +0.600	236	23.6
-0.600 to +0.300	222	22.2
-0.300	64	6.4



Fig: 1 Particle Size Distribution of Coir pith

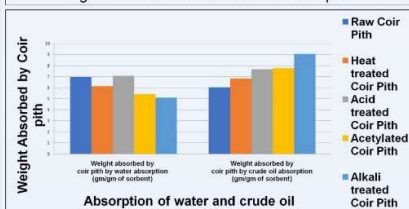


Fig: 2 Absorption of Water and Crude oil by raw & treated coir pith particles

Fig: 3 Absorption of crude oil by Coir dust composite MAT



Fig: 4 SEM image of Untreated & Treated coir composite MAT



CONCLUSION

The permeability behaviors of the coir dust particles and coir dust composite mat were studied and they were sufficient for vertical drains. The permeability reduces in case of coir composite mat.

Characterization of coir pith shows the physical and proximate analysis of coir pith

Particle size distribution of coir pith shows that -1.18 mm to +0.300 mm particles are maximum present in coir pith.

For improving hydrophobicity and oleophilic character of coir pith, Physico-chemical treatments were done and absorption in Water and oil were tested as per ASTM method.

From the SEM Analysis of untreated and treated coir-composite Mat, it is seen that the wax-gum particles were removed in treated of coir dust and the pores of the coir dust were empty. So the absorption capacity of treated coir dust mat increases as compared to untreated coir dust mat.

Product developed for oil absorptions : Coir pith absorbent particles, boards and mats



As coir wastes make some environmental problems, here it is recommended to use them as oil absorbent for prevention of oil spillage, filling material to vertical drains and waste water problems due to its easy modification, abundance and biodegradability

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Figure 28: ESTEC 2020 poster presentation

Paper Publication:

“Development of a high oil absorbent mat from coir dust and evaluation of their physico-chemical properties”, Pallav Saikia, Dipanka Dutta, Dipul Kalita & Jayanta Jyoti Bora (Under review)

Technology Developed: “High Oil Absorbing Mat/Pad from Coir Waste”

1. **Raw Materials :** Coir waste, Natural fibre, Natural binder
2. **Production Capacity :** 60,000 sheets per annum
3. **Application/Uses:**
 - The product is used in oil spilling related to oil exploration, industries, transportation etc.
 - The product can be used in machine/vehicle maintenance work.
4. **Brief technical details:** Absorbing Mat/Pad is made from the mixture of coir waste and natural fibre using a natural binder. The Mat/Pads are used in oil spilling related to oil exploration, industries, transportation etc. Also product can be used in machine/vehicle maintenance work. The product has higher oil absorbing property, reliable tensile strength with flexibility, good tear & smooth surface, better to commercial products.
5. **State of Art:** Coir pith/dust, is one of the solid wastes generated from coir industry and are usually dumped nearby open grounds causing soil and air pollution. These can be converted to value added product by adopted certain mechano-chemical processing. There are some other wastes generated in our day to day activities such as gunny bag/jute waste and waste cotton etc, which can also be effectively utilized with coir wastes for to making useful product. The process for making oil

absorbing Mat/Pad is economic and free from the use of hazardous chemicals. Thus the product is eco- friendly. The use of natural fibre with coir pith/dust in order makes the composite sheet more eco- friendly and durable found suitable for using in oil spilling related to oil exploration, industries, transportation and used in machine/vehicle maintenance work.

6. Innovative Components:

6.1 Patent applied /filed/granted on the technology (if any): 1 Patent applied

6.2 Publication communicated/published on the proposed technology (if any):

Publication communicated: 1 No.

Poster Presentation : 1 No.

6.3 Briefly indicate the innovative component of the process/technology proposed : Oil High oil absorbing Mat/Pad from coir wastes is a new product which is durable and eco- friendly. No effluent/gas generates during the manufacturing of the product. Natural fibre is used to make the product more absorbent, eco-friendly and more durable. A natural binder material used in the process gives higher flexibility, smoothness and more durability.

7. Effluents and the Environmental Consideration: No effluents generates during the manufacturing process. Hence the process is environment friendly.

8. Health consideration and toxicity (if any): The ingredients used in the process are non-toxic. Hence the product doesn't have any negative effect on human being

9. Requirement of approval needed from statutory bodies (if any): --

10. Level/scale of Development : Industrial scale level

11. Industrial Requirements:

11.1 Comments on reproducibility: One time use.

11.2 Thermal and Chemical risk and hazards (if any): Nil

11.3 Safety issues: 100% safe.

Bio-safety considerations, if applicable: Eco-friendly product.

12. Basic raw materials: Coir waste- coir pith/dust, natural fibre and natural binder

13. Major plant and machineries required:

Major plant and machineries required are as follows:

- i. Shedder
- ii. Valley Beater
- iii. Handmade Vat
- iv. Press machine
- v. Weighing balance
- vi. Miscellaneous

14. Cost benefit analysis:

14.1 Unit capacity of the process/technology proposed for transfer: 2 sheets per day or 60,000 sheets per annum

14.2 Tentative project cost for the proposed capacity: Rs. 18.8 Lakhs
(Please see project profile in TABLE-I)

14.3 Profitability: Approx. gross profit 11.95 Lakhs per annum (Please see project profile in TABLE-VI)

14.4 Break-even analysis: 25.64% (Please see project profile in TABLE-VI)

Brief economic impact assessment justifies the proposed technology vis-a-vis present market demand:

High Oil absorbing Mat/Pad is a new product made from coir pith/dust a waste generated from coir industry. Such waste materials are usually damped nearby industrial site which ultimately creates soil or air pollution to a greater extent. The use of such waste material not only reduces environmental pollution but also helps in making high oil absorbing mat/pad for oil spill. Oil spill is one of the major sources that contribute to environmental pollution in oil Industry Due to the gradual increasing environmental pollution in oil industry, hence there is need of control this pollution. The high oil absorbing mat/pad developed by CSIR-NEIST has some speciality for which is suitable for using in oil spilling related to oil exploration, industries, transportation. It can be used in machine/vehicle maintenance work, has also higher market potentiality. Hence, establishment of such industries may create employment opportunity and helps income generation.

15. Brief SWOT analysis of the technology:

Strengths	Weaknesses	Opportunities	Threats
<ul style="list-style-type: none"> • Technology of similar type is presently available at CSIR-NEIST • Low cost, eco-friendly technology • Having expertise in composite making • Availability of machinery and equipment at CSIR-NEIST 	<ul style="list-style-type: none"> • Non availability of sufficient coir waste (Raw material) in the NE region 	<ul style="list-style-type: none"> • The product has high market potentiality • Possibility to set up industry small/medium scale • Availability of natural fibre material 	<ul style="list-style-type: none"> • Raw materials/ Chemicals required for the technology are to be procured from the market • Synthetic absorbent mats are available in the market

PROJECT PROFILE ON HIGH OIL ABSORBING MAT/PAD FROM COIR WASTE

1. <i>Product</i>	:	High Oil Absorbing Mat/Pad From Coir Waste
2. <i>Raw Materials</i>	:	Coir waste, Natural fibre, Natural binder
3. <i>Production Capacity quality</i>	:	60,000 Sheets per annum
4. <i>Project Cost</i>	:	Rs.18.800 Lakhs
5. <i>Break-Even Point</i>	:	25.64 %
6. <i>Know How Fee</i>	:	Rs. 2 Lakhs
7. <i>PI</i>	:	Mr. Jayanta Jyoti Bora ¹
<i>Co- PI</i>	:	Dr. Dipul Kalita ²
<i>Team Members</i>	:	Mr. Pallav Saikia ² Mr. Dipankar Neog ¹ Dr. Tridip Goswami ² Mr. Subodh Chandra Kalita ¹
¹ General Engineering Group, Engineering Sciences & Technology Division		
² Cellulose Pulp & Paper Group, Material Sciences & Technology Division		
CSIR- North East Institute of Science & Technology, Jorhat, Assam, Pin: 785006		

INTRODUCTION

Oil spill is one of the major sources that contribute to environmental pollution in oil Industry. It happens as a consequence of activities related to crude oil exploration, industries, and transportation. On the land, oil spill penetrates to soil matrix and thereby contaminate ground water. The entire process of oil spill pollution is handled worldwide by two ways, restricting oil spill from spreading to the surroundings and removing the spill from contaminated surroundings. Coir pith/dust, a waste generated from coir industry and a natural organic sorbent, is an environmental-friendly, biodegradable, renewable, cheap material and has got excellent chemical and physical properties, which makes it suitable for diverse applications. Since a major portion of this waste is biological origin, hence the use of these materials in making value added products would definitely be helpful from the point of ecology and environment. These solid waste materials alone or in combination with natural fibres may produce useful products through certain mechano- chemical processing. Plant fibres are being used for several generations in man made products/processes due to their easy availability, inexpensive, low density, low energy consumption, biodegradability and renewability nature. There is also a trend that the products made from biological origin are considered to be environmentally benign. They exhibit several advantageous properties like better mechanical strength, lower density, oil absorbing property, yielded light weight flexible composite materials which find use in many industrial applications. Thus incorporating bio-renewable materials makes the final product biodegradable, renewable and more eco-friendly.

A numbers of steps are involved in the preparation of oil absorbent mat using coir pith/dust as raw material. Some important steps include disintegration of the dust materials, mixing with suitable binder, sheets making in handmade vat to get required shape & size, drying of the sheets and final product making.

Considering the gradual increasing environmental pollution by oil spilling in oil Industry, CSIR-NEIST has developed a highly oil absorbing mat/pad from coir industry wastes i.e. coir pith/dust in combination with natural fibre i.e. jute/ cotton fibre using natural binder. Utilization of such waste materials is not only help to reduce environmental pollution, but also help for making a product suitable for using in oil spilling related to oil exploration, industries, transportation and in machine/vehicle maintenance work.

SALIENT FEATURES:

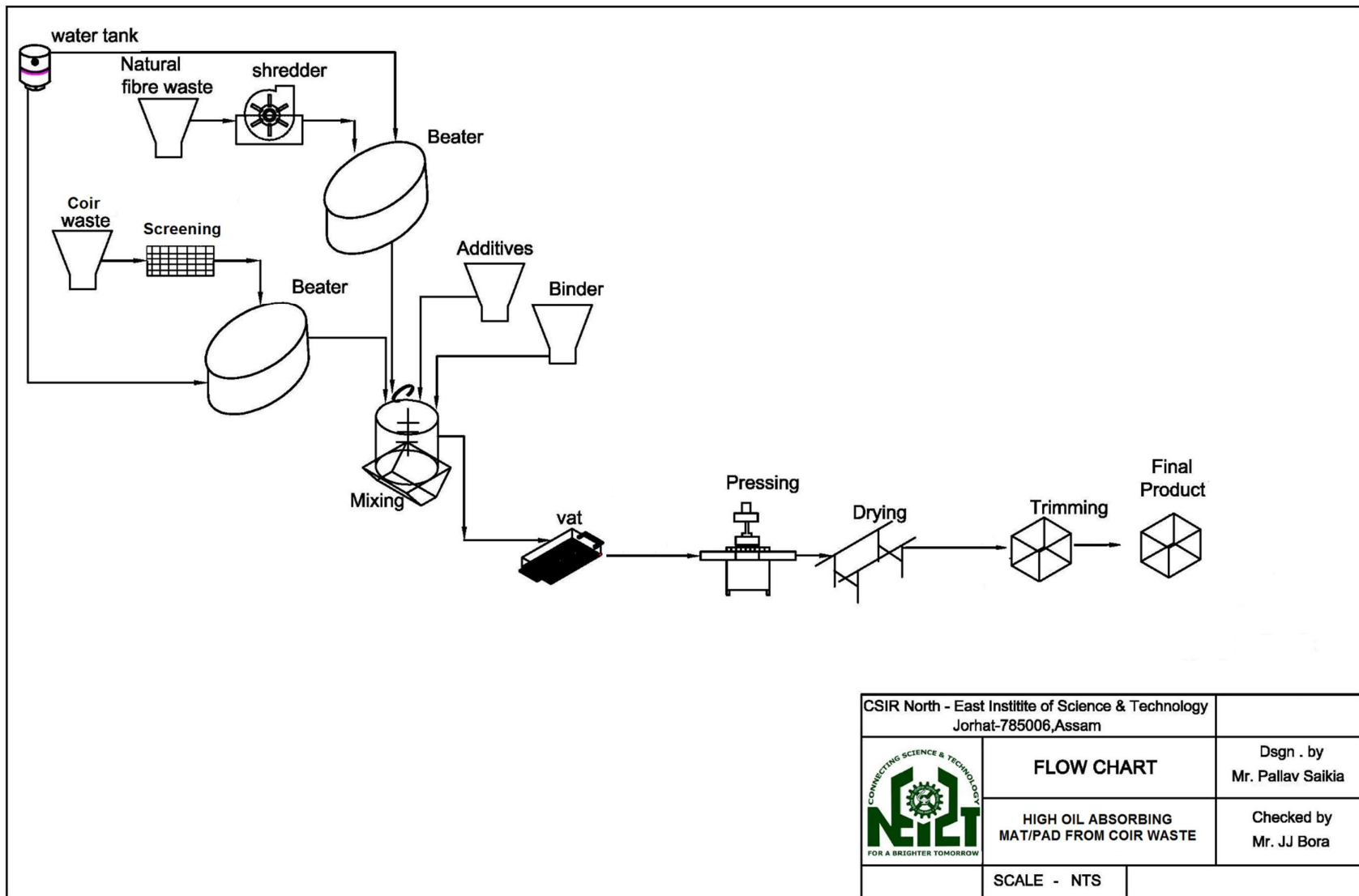
- Use of coir waste and natural fibre for making useful product.
- Recycling of waste material reduces environmental pollution.
- Simple, eco friendly technology with zero discharge.
- Use of solid waste reduces the environment pollution.
- The technology is simple and the final product is eco-friendly.
- Suitable for small/cottage scale industry also.

ADVANTAGES OF THE PRODUCT:

- The product is highly oil absorbing capacity and possesses higher mechanical strength properties.
- Use for oil spilling related to oil exploration, industries, transportation.
- The product is found suitable to use in machine/vehicle maintenance work.
- The product is chipper water repellent, flexible and smooth.

PROCESSING STEPS:

- Cleaning and defibration of the coir waste and natural fibre.
- Mixing and treatment with special chemical of the coir waste and natural fibre.
- Sheet making and pressing of the sheets.
- Sun drying or oven drying of the sheets.
- Trimming and packaging of finished product.



USES:

- The product is used in oil spilling related to oil exploration, industries, transportation etc.
- The product can be used in machine/vehicle maintenance work.

TECHNO- ECONOMIC:

The investment cost for production of 200 sheets per day capacity plan will be approximately Rs. 18.80 Lakhs.

TABLE- I
PROJECT CAPITAL COST

Sl. No.	PARTICULARS	Amount (Rs.)
A	MACHINERY	12,95,000.00
	Shredder, Valley Beater, Handmade Vat, Screw press/Hydraulic press, Weight Balance, Miscellaneous, Electrical Fitting & Lighting, Office furniture	
B	PRE-OPERATIVE EXPENSES	50,000.00
C	MARGIN MONEY FOR WORKING CAPITAL	3,10,000.00
D	KNOW HOW FEE + CONSULTANCY FEE	2,00,000.00
E	GST 12% of D	24,000.00
GRAND TOTAL		18,79,000.00

TABLE - II

COST OF PRODUCTION

Number of Working Days =300

Plant Capacity = 200 Sheets per Day

60000 Sheets per annum

Sl. No	Item	Requirement		Price (Rs)		Amount (Rs)
1	Raw Material Cost					
	Coir Pith	92.79	Kg per day	8.00	per Kg	2,22,693.82
	Natural fibre	16.37	Kg per day	30.00	per Kg	1,47,370.91
	Binder	10.92	Kg per day	100.00	per Kg	3,27,490.91
	Beater Additive	1.20	Kg per day	6.00	per Kg	1,440.96
	Raw Material Cost					6,98,996.60
2	Manpower Cost	As per Table III				6,18,000.00
3	Utility					
	Electricity	35	units per day	5.71	per unit	59,955.00
	Water	1000	litre per day	2.50	per 100 litre	7,500.00
	Utility Cost					67,455.00
4	Packaging Cost	200	sheets per day	0.25	per sheet	15,000.00
5	Maintenance & Repair	5%	of Machinery			64,750.00
6	Depreciation	10%	of Machinery			1,29,500.00
	Depreciation Cost					1,29,500.00
7	Interest on Bank Loan	10.5 %	of Bank Loan			1,97,295.00
8	Miscellaneous Expenditure	2%	of Raw material Cost			13,979.93
TOTAL COST OF PRODUCTION						18,04,976.53

SALES REALIZATION

Sl. No	Item	Quantity		Selling Price (Rs)	Amount per Annum (Rs)
1	Oil Absorbent Mat/Pat	200	Sheets per Day	50.00 per sheet	30,00,000.00
TOTAL SALES REALIZATION					30,00,000.00

TABLE - III

Cost of Manpower

Sl. No.	Type	Quantity	Wage/ Monthly Salary in Rs.	No. of Days or month		Amount in Rupees
1	Production Manager	1	9000.00	12	months	108000.00
4	Labour	6	250.00	300	days	450000.00
5	Watchman	1	5000.00	12	months	60000.00
Total Yearly Cost						618000.00

Table - IV

Margin Money for working capital

Sl. No.	Particulars	No. of Months		Amount in Rupees
1	Raw Materials	2	Months	116499.43
2	Manpower Cost	2	Months	103000.00
4	Utility	2	Months	11242.50
5	Packaging Cost	90	days	4500.00
5	Maintenance & Repair	2	Months	10791.67
6	Depreciation Cost	2	Months	21583.33
7	Interest on Bank Loan	2	Months	32882.50
8	Miscellaneous Expenditure	2	Months	2329.99
TOTAL				302829.00
Say				310000.00

PROFITABILITY ANALYSIS

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TABLE - VI

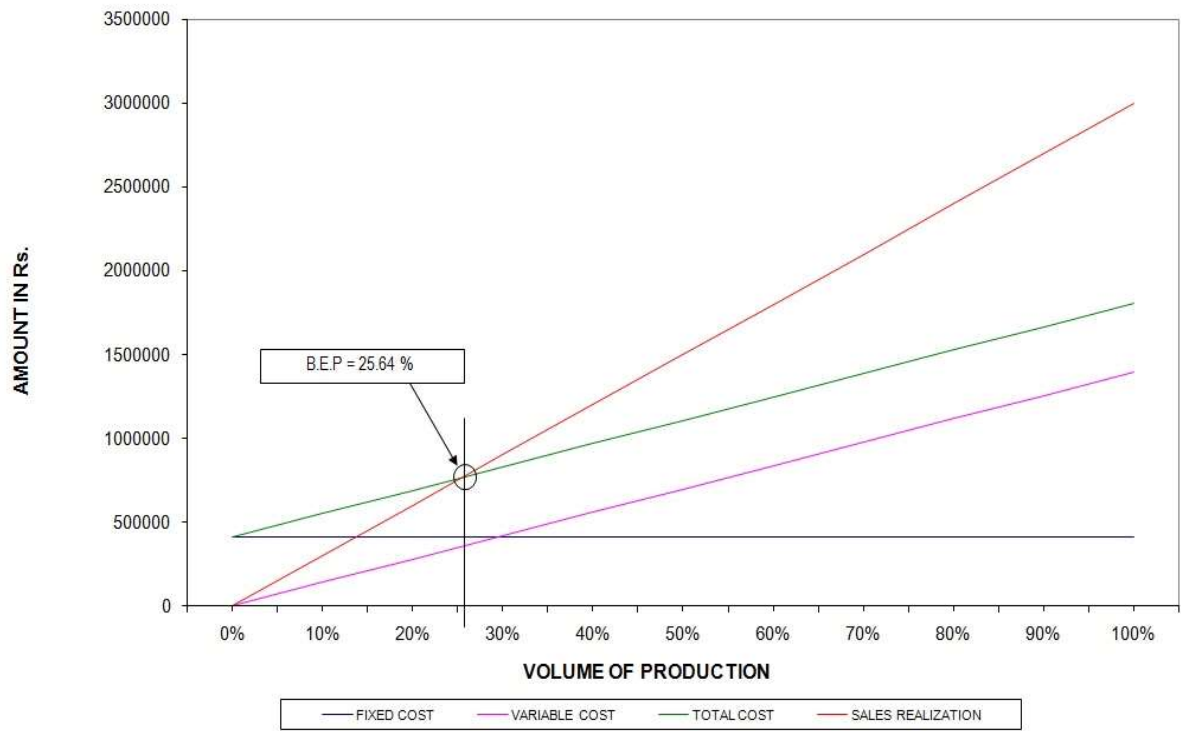
BREAK-EVEN ANALYSIS

Sl. No.	Particulars	Amount in Rupees
A	FIXED COST	
1	Manpower Cost	60000.00
2	Interest on Bank Loan	197295.00
3	Utility	9542.39
4	Depreciation	129500.00
5	Maintenance & Repair	12950.00
6	Miscellaneous Expenditure	2795.99
	TOTAL OF (A)	412083.37
B	VARIABLE COST	
1	Raw Material Cost	698996.60
2	Manpower Cost	558000.00
3	Utility	57912.61
4	Packing Cost	15000.00
5	Maintenance & Repair	51800.00
6	Miscellaneous Expenditure	11183.95
	TOTAL OF (B)	1392893.15
C	TOTAL COST (A + B)	1804976.53
D	SALES REALIZATION	3000000.00
E	GROSS PROFIT	1195023.47
F	BREAK - EVEN POINT	25.64%

COST OF PRODUCTION PER SHEET 30.10

SELLING PRICE PER SHEET 50.00

BREAK-EVEN ANALYSIS





High Oil Absorbing Mat/Pad



Honourable Union Minister of Road Transport & Highways and MSME, Govt. Of India Sri Nitin Jairam Gadkari launched technology on **Oil Absorbing Mat from Coir Pith** which is developed under Collaborative Project “**Development of Appropriate Product by Studying the Possible Use of Coir Dust in Oil Industries with Reference to North-East India for Absorption of Oil Spill**” of CSIR-NEIST & COIR BOARD on 28th September, 2019 in the Inaugural function on the new initiatives of COIR BOARD at Vellore, Tamilnadu.

FINAL PROJECT REPORT

(Period: January, 2016 - July, 2019)

Prepared by CSIR-NEIST, Jorhat

Project Title:

*Development of Appropriate Product by Studying the
Possible Use of Coir Dust in Oil Industries with
Reference to North-East India for Absorption of Oil Spill.*

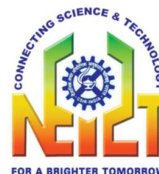
A Collaborative project from

COIR BOARD

(Ministry of Micro, Small and Medium Enterprises)

Govt. of India

Kalavoor – 688522, Alleppey



उत्तर-पूर्व विज्ञान तथा प्रौद्योगिकी संस्थान

CSIR-North East Institute of Science and Technology,

Jorhat – 785006, Assam

INDIA

Connecting Science & Technology for a Brighter Tomorrow

ACKNOWLEDGEMENT

I am highly grateful to Coir Board (Min. of MSME) Govt. of India for financial support to carry out the research work. Also, I am indebted to the Director, CSIR-NEIST Jorhat for his keen interest and necessary support to carry out the work successfully in the institutes. My sincere thanks to Co-PI: Mr Dipankar Neog, Members: Mr. Dipul Kalita, Mr. Dhanjit Das, Dr. R. L. Goswami, Technical staff and Research scholars Mr. Bishnu Sahu, Ms. Maitreyee Chakrabarty (General Engineering Group) & Mr. Pallav Saikia (CPP Group) of the Institute for their help and kind co-operation for successful completion of the project.

(Mr. Jayanta Jyoti. Bora)

Principal Investigator

COMPLETION REPORT OF EXTERNALLY FUNDED PROJECTS (2016-2019)

Non Technical Details

1. Title of the Project:
"Development of appropriate product by studying the possible use of Coir Dust in Oil Industries with reference to North-East India for absorption of oil spill".
2. Reference:
Project Sanction No. CCRI/Res/CP-NER-I/2015-16/15/42/1561 dated 04/01/2016
3. Duration of Project:
Submitted Period: 3 Years 6 months (January 2016 to July 2019)
4. Date of commencement of the Project: 13/01/2016
5. Date of completion of the Project: 12/07/2019
6. Funding Agency: Coir Board (Min. of MSME) Govt. of India
7. Total Cost (Rs.): Rs. 53.13 Lakhs (Total Amount Received is Rs. 50.47350 lakhs)
8. Principal Investigator (Name and Address)
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General Engineering Group (ESTD)
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Jorhat-785006, Assam
- 8.1 Co- Principal Investigator (Name and Address)
Mr. Dipankar Neog, Pr. Scientist.
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EXECUTIVE SUMMARY

The present research successfully explored the possibilities of utilizing coir dust, a waste product generating from the coconut husk and fibres, as development of a useful product that could use for clean-up oil pollution i.e. Oil spillage which is the major sources that contribute to environment pollution in oil industries. The Studies undertaken in the project to developed product i.e. Oil Absorbent Particles, Oil Absorbent Mat/sheet, Oil Absorbent block/ bricks/ board of those waste for defend the soil from oil spill and also minimize the coir waste material.

The project works studies the characterisation of coir dust i.e. Proximate analysis, lignin content, cellulose content, pH value, physico-chemical treatments etc. were done to enhance the hydrophobicity and oleophilic character of the coir dust. Coir dust was then exposing to water and crude oil to determine hydrophobicity and oil sorption capacity. After that application in industrial product form, design and fabricate waste coir dust as Oil Absorbent Mat (OAM) which is made from coir dust (CD) in combination with commonly available natural fibre i.e. cotton waste (CW) and jute waste (JW) in the ratio 80:20, 85:15, 90:10. The physical strength of the mats were found to be maximum at 85:15, having tensile strength 43.86 ± 3.5 and 49.17 ± 4.0 MPa for composite of CD and CW, and CD & JW respectively. Similarly, oil absorption also recorded 21.8 ± 2.0 and 19.6 ± 2.0 g/g in crude oil. Various mechanical properties of the product, for instance oil absorption, double fold, bursting strength, ultimate tensile strength (UTS) etc. were determined along with thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). CD, CW and JW were characterized by FTIR, XRD, TGA and SEM analysis. Results clearly portray that natural fibre significantly improves the oil absorption, mechanical and thermal properties, and also increases the flexibility of the final composite mat. These mats are an advantage over the commercial ones in terms of higher oil absorbency and physical strength.

Chapter 1

Introduction

Review of Literature:

Oil spill is one of the major sources that contribute to environmental pollution in oil Industry. It happens as a consequence of activities related to crude oil exploration, industries, and transportation. On the land, oil spill penetrates to soil matrix and thereby contaminate ground water [1]. The entire process of oil spill pollution is handled worldwide by two ways, restricting oil spill from spreading to the surroundings and removing the spill from contaminated surroundings. Although, petrochemical plants and oil refineries are helpful to society, but they produce a large amount of hazardous waste [2]. Additionally, oil spills during exploration, transportation, and refining, have caused serious environmental problems [3]. Regular physico-chemical methods can quickly remove the majority of spilled oil, but some simply transfers contaminants from one environmental medium to another medium and produce toxic by-products. Moreover, crude oil cannot be completely cleaned up with physicochemical methods [3].

A huge quantity of coir dust i.e. a major by-product is produced, in the processing of coir fibre from the coconut husk and development of product from the processing fibre [4]. Annually around 7.5 million tons coir dust/pith is produced [5]. Increasing of coir waste in the surrounding area of coir industries day by day, has created two vital problems (i) occupied valuable ground space, and (ii) contamination of potable groundwater due to percolation of remaining phenolics from these dumps [5]. It decomposes very slowly in soil as its pentosan-lignin ratio is below 0.5 [6], and due to chemical and structural complexity of lignin cellulose complex [7]. CD is mentioned as that brownish colour with light weight spongy particle which falls out at the time of fibre processing [8]. About 70% of the weight of the coconut husk is coir dust [9]. This coir dust has been disposal by the

burning or dumped without control, otherwise use at crop plant as raw organic manure [10-12]. This burning also creates various environmental problems. It contains higher amounts of cellulose in addition potash and lignin and ability to excellent moisture persevering capacity but decomposition is time-consuming [13]. It's have no any commercial value except, may be, in applications where sawdust is used in a very limited amount. Coir dust, a good oil absorbent media, shows superior quality result the oil absorbency [14]. Moreover, Coir pith or dust provides perfect oil absorption medium when, used in small particulate sizes [14]. Coir pith or dust is used to clean up oil spills in solid surfaces, highways, concrete slabs, soils, as well as the surfaces of water bodies [14]. It is reported to be composed of cellulose, pentosan, furfural, and lignin with excellent moisture retaining capacity, but is slow in decomposition [13, 15, 16]. However, it is necessary to find an instant solution to the constant problem of coir dust disposal by proper utilization of this waste material.

Since a major portion of this waste is of biological origin, hence the use of these materials in making fruitful products would definitely be supportive from the point of ecosystem restoration. These coir waste materials alone or in combination with natural fibres may produce useful products through certain mechano-chemical processing. Due to the nature of easy availability, cheap, high oil absorption capacity, lower density, minimum energy utilization, biodegradability and renewability of natural fibres, several generations are using it in synthetic products [17]. Generally, the products manufactured from biological source are considered to be environmentally benign [18]. They show various beneficial properties like superior oil absorbing property, physical strength, lower density, which yield light weight absorbent mats that find use in many industrial applications. Natural fibres are also utilized in thermoplastic polymers reinforcement for automotive applications as well as construction materials etc [19-21]. Thus incorporating bio-

renewable materials make the finished product higher oil absorbent, biodegradable, renewable and more eco-friendly.

For effectual use of such industrial waste, a study was undertaken to develop high oil absorbing mat/pad from coir dust in combination with plant fibres like cotton and jute wastes, implementing a simple process using commercially available equipments. The cotton fibre is an ideal oil-absorbing material due to its low density and sloppy internal structure as well as great fluid adsorption space [22]. The physico-mechanical strength properties like UTS, folding endurance, bursting, density as well as oil absorption properties of the final product and the raw materials used, i.e. coir dust, cotton and jute, were determined as per American Society for Testing and Materials (ASTM) and Indian Standard (IS) methods. The OAM and the raw materials were also characterized by FTIR, XRD, TGA and SEM.

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Chapter 2

Objectives and experimental outlines

2.1 Objectives:

1. Study on the characterisation of coir pith such as calorific value, moisture content, ash content, cellulose content, lignin content, ph value, alkali solubility (1 % NaOH solution), cold water solubility, hot water solubility, bulk density, porosity, water holding capacity, Swelling % etc according to standard methods (TAPPI, 1980; AOAC, 1975).
2. Study different Physico-chemical treatments of coir pith as an absorbent to be used for prevention of oil spillage effect with reference to NE oil industries.
3. For improving hydrophobicity and oleophilic character of coir pith, Physico-chemical treatments such as heating at relative low temperature, acid treatment, alkali treatment, acetylation and acylation treatment were done and absorption in Water and oil were tested as per ASTM method. The properties of treated coir pith were characterized by FTIR and SEM.
4. Design and development of coir dust sorbent in the shape of membrane/ blanket/ block/ granules/ pad/ Sheet to deal with oil spillage.
5. Mechanical properties and oil sorption capacity of the developed Mat/ Sheet/ granules/ bags were done. Also comparison between various oil absorbent and oils were done and sent for field trial.
6. Study on possibilities of re-use & disposal of coir dust sorbent.

The whole research work of the project has been conducted at CSIR-North East Institute of Science and Technology

2.2 Experimental Outlines:

The physico-chemical properties of coir dust were evaluated as per TAPPI Standard Test Method and ASTM method. The Coir dust were suitably treated (i) chemically, (ii) physically and (iii) a suitably combination with other natural organic waste such as cotton waste and Jute waste thereof and the effect of such treatment on properties of coir dust were studied and compared with the untreated Coir dust. The downsizing of coir dust was done mechanically to generate particles of variant sizes which were subsequently separated into different sizes (mesh sizes) by screening process (sieve shaking). The composite boards were prepared by mixing of coir dust in the form of particles with binder under conditions of hydraulic hot pressing. The composite boards were characterized in terms of physical properties (density, water absorbency, oil absorbency, thickness etc.) and mechanical properties (tensile strength, breaking load, etc.). The process of composite board making was studied via examination of influence of different process variables (weight ratio of particle to time, temperature, pressure and binders (chemical agent)) on the properties of composite boards. The usefulness of the composite boards as oil absorbent was tested. Similarly the composite Oil absorbent Mat (OAM) has been developed using coir waste material, such that soil pollution from oil seepage into the soil from coir dust may be minimized. OAM is made from coir dust (CD) in combination with commonly available natural fibre i.e. cotton waste (CW) and jute waste (JW) in the ratios of 80:20, 85:15, 90:10 respectively. Various mechanical properties of the product, for instance oil absorption, double fold, bursting strength, ultimate tensile strength (UTS) etc. were determined along with thermogravimetric analysis (TGA) and scanning electron microscopy (SEM). CD, CW and JW were characterized by FTIR, XRD, TGA and SEM analysis.

Chapter 3

Materials and Methods

3.1 Collection of coir pith and crude oil:

3.1.1 Collection of coir pith from different coir industry:

The coir pith was purchased from Coir Industry, Sonitpur (Biswanath Chariali source), Assam and was supplied by Amrit Organics, Duliajan Assam. Coir pith samples from Nalbari source, Assam have also been purchased for a comparative study. After collection, the moisture content of the Coir dust was determined in a laboratory moisture meter. The Coir dust (CD) were screened and then washed properly with cold fresh water and sun dried. The dried CD were packed in polythene bags and kept for subsequent study.

3.1.2 Collection of Crude oil samples:

Collection of crude oil samples were from

- a) Oil India Ltd., Duliajan, Assam and
- b) Pumping Station (PS-3), Oil India Ltd., Charigaon, Jorhat, Assam.

3.2 Proximate Chemical Analysis/ characterisation of Coir dust:

3.2.1 Determination of Ash Content:

According to the TAPPI standard method T-211 cm-86 (TAPPI, 1980), Coconut coir dust was air dried for 24hrs in oven at 60°C to constant weight. Coir dust (1g) was weighed in a Platinum crucible and then the crucibles were put into a muffle furnace at 575±25°C. After ignition for 4 hours, the crucible (with washed and unwashed sample) were cooled slightly and placed in desiccators. When it cooled to room

temperature, weighed the ignited crucible on an analytical balance to the nearest 0.1 mg. The results were expressed as % of the moisture free coir dust as follows:

$$\text{Ash content (\%)} = A/B * 100$$

Where, A= OD weight of Ash in g.

B= OD weight of the test specimen in g.

3.2.2 Determination of calorific value of coir dust:

The calorific value of coir dust was determined by taking two samples of coir dust (washed and unwashed) of two sources in an Automatic Bomb Calorimeter (Leco, AC-350, Model No. : 603-300-100).

3.2.3 Determination of Moisture Content in Coir Dust:

The unwashed coir dust (1g) was measured into a pre-weighed porcelain dish with cover and weighed. The coir dust and the container with lid open was placed in an oven at 60⁰ C for 5 hours. Further drying was carried out at 80⁰ C for 3 hours. The container with the lid was allowed to cool and reweighed with minimum exposure to atmosphere. The last drying was repeated for another 3 hours till a constant weight was obtained. ^[47] Moisture content determined according to the formula:

$$\text{Moisture Content (\%)} = [(W_1 - W_2) / W_1] * 100,$$

Where, W₁ is the initial weight of coir dust before drying and W₂ is the final weight of coir dust after drying.

3.2.4 Solubility in Cold Water:

Solubility of coir dust in water is determined by the TAPPI standard method T- 207 m-54 (TAPPI, 1980). 1 g of coir sample is digested at room temperature with 300 ml of distilled water with frequent stirring for 48 h. The mixture is then filtered and residue is washed with cold distilled water and then dried at 100⁰ C \pm 5 °C and

weighed in a stopper weighing bottle to constant weight. The loss in weight of the substance is calculated in Percentage as cold water soluble material.

$$\text{Cold water solubility (\%)} = [(A-B) * 100] / A$$

Where, A = Initial OD weight of the specimen in g.

B = OD weight of the specimen after extraction in g.

3.2.5 Solubility in Hot Water:

According to TAPPI standard method, T-207 m-54 (TAPPI, 1980), 1 g of coir sample is digested at with 100 ml of distilled water in a water bath under reflux condenser for 3 h. The mixture is then filtered and residue is washed with hot distilled water and then dried at 100-105°C to constant weight. The loss in weight of the substance is calculated in Percentage as hot water soluble material.

$$\text{Hot water solubility (\%)} = [(A-B) * 100] / A$$

Where, A = Initial OD weight of the specimen in g.

B = OD weight of the specimen after extraction in g.

3.2.6 Solubility in Dilute Alkali:

According to TAPPI standard method, T-207 m-54 (TAPPI, 1980), Oven dried coir dust (1g) is stirred with 100ml of 1% solution of NaOH, in covered beaker, which is placed in a boiling water bath for exactly 1 h with intermediate stirring at 10, 15 and 25 minutes. The mixture is then filtered by suction on a tarred crucible and the residue is washed in succession with hot water, 50 ml of 10% acetic acid and again with hot water. The final residue is then dried at 100-105 °C and weighed. The result is expressed as percentage on the oven dry weight of coir dust.

$$\text{Dilute Alkali solubility (\%)} = [(A-B) * 100] / A$$

Where, A = Initial OD weight of the specimen in g.

B = OD weight of the specimen after extraction in g.

3.2.7 Determination of cellulose content:

2 g of coir dust was taken in a conical flask; 10 ml of bleach (Sodium hypochlorite) is added in 90 ml of deionised water into the conical flask. The mixture was boiled until sample is bleached. It is then filtered, washed and dried at 80°C. To the bleached sample, 100 ml of 24 % aqueous NaOH is added and boiled for 1 hr to remove hemicelluloses. Then residue was filtered, washed and dried at 80 °C for 2-3 hours for getting constant weight for calculation. Calculation was made following the relation given below.

$$\text{Cellulose content (\%)} = A * 100 / B$$

Where, A = OD weight of cellulose in g.

B = OD weight of the test specimen taken in g.

3.2.8 Determination of Acid insoluble Lignin content:

According to TAPPI standard method T-13 m-54 (1980) 1g of the sample was taken, mixed with 15 ml of 72 % H₂SO₄ at 20 ± 1°C and kept in a water bath for 2 hr maintaining constant temperature 20 ± 1°C. Transferred the material to a beaker and added water so that total volume becomes 575 ml. Boiled the Solution for 4 hrs in a round bottom flask with 1m long glass pipe joint so that the vapours are cooled in pipe and come down and there is no loss. Keep the round bottom flask for overnight to settle the insoluble material. The final residue was then filtered, washed, dried at 105 ± 3°C and weighed. The result was expressed as percentage as follows.

$$\text{Lignin content (\%)} = A * 100 / B$$

Where, A = OD weight of acid insoluble lignin in g.

B = OD weight of the test specimen taken in g.

3.2.9 Determination of pH of coir dust in water:

1 g of dry coir dust (washed and unwashed each) was added to 30 ml tap water and allows soaking for 15 minutes. After that squeeze the coir dust from water and measure the pH of the each solution with pen type pH meter. It was found that pH for washed coir dust was 6.64 and unwashed was 6.90.

3.3 Physical properties of Coir dust:

3.3.1 Determination of percent swelling of coir dust in water:

Percent swelling was determine gravimetrically by swelling a sample of coir dust in deionised water and determine weight of the swollen sample and the weight of the corresponding dry sample. Coir dust 1 g was taken in 100 ml of deionised water in a conical flask for 1 hour. The weight of the swollen sample was determined and weight of the dried sample determined.

$$\% \text{ Swelling} = A * 100 / B$$

Where, A = weight of the swollen coir dust

B = weight of the dry coir dust

3.3.2 Determination of Bulk Density of coir dust:

We take a beaker and determine its volume V and its weight W₁. Then we fill the beaker with coir dust and take its weight W₂.

$$\text{Bulk density} = (W_2 - W_1) / V$$

3.3.3 Determination of Total porosity, Aeration porosity and Water holding porosity of Coir dust:

In order to determine the porosity, a plastic cylindrical container of 250 ml capacity was taken. The bottom of the container was pierced with a fine needle so as to have 10 holes uniformly distributed at the bottom of it. The 10 holes were then closed with a waterproof adhesive tape. Then the container was filled with the coir dust by gently tapping it till the coir dust fills the 250 ml mark. The water was slowly dripped over the coir pith so that the coir pith was completely drenched and saturated. This process took several hours. The total volume of water added (A) in ml was recorded. Then the container was placed over a water proof pan and the adhesive tape at the bottom was removed so as to drain the water on the pan. Then the drain water (B) in ml was measured.

$$\text{Total porosity \%} = A * 100 / 250$$

$$\text{Aeration Porosity \%} = B * 100 / 250$$

$$\text{Water holding porosity} = \text{Total porosity} - \text{Aeration porosity}$$

3.3.4 Determination of percent weight of graded Coir dust:

1000 g of Coir dust sample is taken for sieve analysis to assess the particle size distribution. From the distribution it was seen that $> 1.18 \text{ mm}$ to $< 600 \mu\text{m}$ size particles were present in highest %. Also particle size between 2.36 mm to 1.18 mm and $600 \mu\text{m}$ to $300 \mu\text{m}$ were mostly presents in the graded coir dust.

3.4 Physico-Chemical Treatment of coir dust:

3.4.1 Heat treatment of coir dust at relatively low temperature:

Heat treatment was done by heating 15 g of coir dust at 150°C for 17 minutes in hot air oven. This treatment of coir dust was done to improve its hydrophobicity.^[45]

After that it was exposed to water and oil to determine the hydrophobic and oil sorption capacity.

3.4.2 Acetylation treatment of coir dust:

1 g pre-treated (washed and dried) coir pith of 600 μm was placed in a 250 ml R.B. flask containing 20 ml of acetic anhydride solution and 0.05 g DMAP. The flask was heated at 120 $^{\circ}\text{C}$ for 30 minutes with reflux condenser fitted. After that the coir dust was washed by ethanol and acetone to remove the unreacted acetic anhydride and acetic acid by product. The product was then dried at 50 $^{\circ}\text{C}$ for 16 hours in hot air oven.^[45] Acetylated coir product was carried out for absorption test.

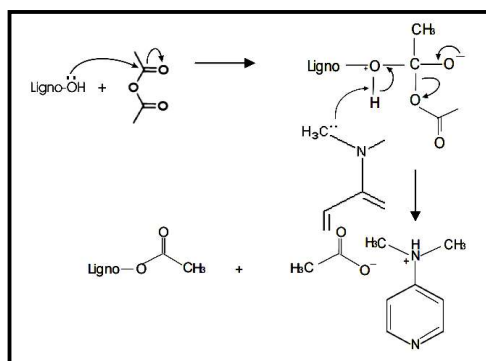


Figure: The mechanism of Acetylation reaction on Lignocelluloses

3.4.3 Acid treatment of coir dust:

We have done acid treatment of 600 μm coir dust particles by mineral acid. Acid treated coir pith was carried out for absorption test.

3.4.4 Alkali treatment of coir dust:

10 g of washed and dried coir dust was taken in a R.B. flask and added 300 ml of 5 % NaOH to it. Heat the mixture at 100 $^{\circ}\text{C}$ for 3 hours. After that the mixture was cool for whole night. Washed it properly with distilled water and dried for 5 hours at

60 °C in hot air oven. After that weight of the alkali treated coir dust was taken and it was found that 85.22 % of coir dust was digested. Alkali treated coir dust was exposed to oil and water for sorption test.

3.4.5 Esterification of coir dust with fatty acid chloride (Acylation treatment):

Bio sorbent (Coir dust) Preparation:

The coconut coir raw material used in this study originated from Biswanath Chariali source, Assam. The raw coir pith was ground into small and short shapes in a high speed grinder, and the visible dust/impurities were removed by washing and boiling with distilled water. Following this, the coir pith was washed several times with acetone before drying in the oven at 60 °C overnight. To ensure the freshness of the raw materials, no further treatment was carried out. ^[46]

Esterification Process:

The Esterification reaction of coir was carried out in a reflux setup maintained under a fume hood. For that, approximately 5 g of coir pith was weighed and placed in a 250 ml two necked round bottom flask. To this, 100 ml of 1% (v/v) N-bromosuccinimide (NBS) catalyst prepared in a DMAc / LiCl solvent system was added and heated up to 100 °C with stirring. To this mixture, 10 % (v/v) concentration of Oleoyl chloride was added drop-wise, and the reaction was allowed to proceed continuously for 4 hours under the same conditions of temperature and stirring. After the period during which the Esterification process was completed, the Acylated coir dust was filtered and washed with a series of solvents including toluene, ethanol, water, and acetone to remove any unreacted oleoyl chloride and unwanted by-products. Then the coir dust was dried to a constant weight in an oven at 70 °C.^[46] The same synthetic process was repeated

for 20 % & 30 % oleoyl chloride are done. Absorption test in Crude oil were done for the same.

3.5 FTIR study of treated coir dust:

The coir dust was evaluated by FT-IR. FT-IR studies were conducted by using IR affinity-1, Shimadzu, Japan; FT-IR Spectrophotometer.

3.6 Composite board making:

Boards were made in the laboratory using untreated and chemically treated Coir dust and different binders (LDPE, Starch & Foaming Agent). We make the blocks of coir dust from two sources (Biswanath Chariali coir dust & Nalbari Coir dust) mixed with each binders in such a way that these were uniformly distributed all around the Coir dust mass. The mixture was put into the wooden mould size 30 × 30 cm and hydraulic hot pressed at 140 ± 2 °C for 20-30 minutes and at 2 kg/cm² pressure. After that, the pressure was released from the hydraulic hot press and the board was kept for some time in open air for conditioning. The properties of the boards made from each treated coir dust and binders were studied. The desirable characteristic in the block was stability and high absorption rate.

3.7 Testing of Composite board Samples (Mechanical & Oil Sorption):

Tensile strength of composite specimens was analyzed at room temperature and 55% RH using Universal Testing Machine (UTM) INSTRON Make, Model 5594. Ultimate tensile strength, maximum load, tensile modulus values were calculated by the software Merlin software version V22054. The values of elongation at break were calculated using equation:

$$\text{Elongation at break (\%)} = \text{Tensile Strength/Tensile Modulus} \times 100$$

For determination of Modulus of Rupture (MOR), 3 point flexural test attachment was used. The MOR was then calculated and expressed in N/mm² by equation:

$$R = 3PL / 2bd^2$$

Where,

P - Maximum load in kg

L - Length of span in cm

b - Width of specimen in cm

d - Depth of specimen in cm

Absorption rate of composite coir dust board were checked in crude oil by cutting the composite boards into small blocks in different interval of times. Then calculate the weight gained by the blocks and graphs were representing with respect to time vs. weight gained.

3.8 Development of Coir dust Bag for Absorption Test in crude oil:

To use Coir dust practically for prevention of oil spillage in the oil fields and other affected areas we have prepared coir pith bags/ pouches. We make bags of markin cloth for containing treated coir pith and check its absorption rate in crude oil.

3.9 Development of coir dust handmade paper (round) for absorption test in crude oil:

Procedure: Coir dust was taken and beat it in the beating machine for 2-3 hours. Similarly cotton waste were beat in the same process to make pulp. After that collected the samples and mixed them in specific ratio (10% & 20% Cotton waste) and make sheet of the same in the Sheet form machine. Then the sheet formed was pressed in the screw pressed machine such that it releases the water from the pulp

sheet. After that dried it for the day and exposed to oil and water to check its sorption capacity

3.10 Development of coir dust various treated handmade paper for absorption test in crude oil:

We made various treated coir dust composite handmade paper. Those were Oven dried coir dust paper; Hot plate press handmade coir pith paper; Oil treated handmade coir pith paper and Latea treated handmade coir pith paper. Expose those papers to oil and water for absorption.

3.11 Development of a high oil absorbent mat from coir dust and evaluation of their Physico-chemical properties:

Coir dusts were collected from North East Coconut Products (P) Ltd., Assam, India, which were initially screened to carry out the experimental work. Natural fibres i.e. cotton waste (hosiery cuttings) and jute waste (gunny bag cuttings) were collected from nearby local market of Jorhat, India and cut initially into the size ranged between 1.5-2.0 cm length to carry out all the experiments. All the chemicals used in the experimental work were procured from Spectrochem, India and used without any purification.

Proximate chemical constituents of coir dust, jute and cotton waste were carried out using the analytical method suggested by Technical Association of Pulp and Paper Industry (TAPPI, USA). For determination of chemical constituents, the CD powder, jute and cotton waste samples were cut into 1.5-2.0 cm length and dried in oven for a period of 6-7 h at 90 ± 5 °C and then powdered in a Wiley mill. The powdered fraction was screened with 40 and 60 BSS mesh and the fraction passed through 40 BSS mesh and retained on 60 BSS mesh (+40 -60) was taken for constitutional analysis.

Lignin content of plant materials was determined by TAPPI, T-222 om-83 and cellulose content by a method of suggested by S. K. Thimmaiah [23]. FTIR spectra (4000-500 cm⁻¹) were recorded in a Shimadzu IR Affinity-1 spectrophotometer on KBr discs. Thermal gravimetric were carried out using TA Instrument (SDT Q600). Under a nitrogen environment flow of 100 ml/min, the samples were heated from 20 °C to 700 °C at a heating rate of 10 °C/min. Scanning Electron Microscopy of CD, NF and OAM were carried by Leo 1430 vp operated at 3 KV on gold coated sample and images were collected at different magnifications. Powder XRD diffractions were carried out on a Rigaku, Ultima IV X-ray diffractometer from 2-80° 2θ, using CuKα source (λ=1.54056 Å). The crystallinity index (CI) was analysed using Equation 1, where I₀₀₂ is the maximum intensity of the I₀₀₂ lattice reflection and I₁₀₁ is the maximum intensity of X-ray scattering broad band, because of amorphous region of the sample [24].

$$CI (\%) = \frac{I_{002} - I_{101}}{I_{002}} \times 100 \quad \dots\dots\dots 1$$

Tensile strength of CD, NF and OAM were determined at 25 °C and 55 % RH using Universal Testing Machine, Make TWI, Model TUTE 10T and values were also calculated by the UTM10 DCCP TWI LCD software version SP2 PACK 04/13. According to ASTM D6182 test method, folding endurance strength was also determined using double fold tester, MIT type machine, Model UEC-1007-C. With the help of Bursting strength tester, Model UEC-1010-B1, bursting strength carried out according to ASTM D2207 test method. Oil absorption test for the oil absorbent mat using different oils (Crude Oil, Diesel Oil, Hydraulic Oil, Transformer Oil, Mobil Oil) at 20- 30 °C was conducted following IS specification [25].

3.11.1 Preparation of Oil Absorbent Mat (OAM) sample:

Coir dust, Cotton waste i.e. hosiery cuttings and jute waste i.e. waste gunny bag cuttings were samples were beating separately with a laboratory beater for 3 h using deionised water at 1.5 % uniformity. Two separate stocks prepared from coir dust, cotton waste/ jute waste were supplemented with 4 % guar gum and 1 % alum during stock preparation. Absorbing mat were made in the handmade vat, keeping coir dust waste to jute/cotton at 80:20, 85:15, 90:10 ratio, followed by screw press machine to eliminate the surplus water from the wet mat/pad and then drying of mats.

Chapter 4

Results and Discussions

4.1 Proximate Chemical Analysis/ Characterisation of coir dust:

The results of proximate chemical analysis/ characterisation of coir dust are shown in Table 1. The study shows that there are lots of soluble materials in Raw Coir dust. Cellulose content were found to be 35.90 % (Nalbari Source coir dust) and 36.56 % (Biswanath Chariali Source coir dust), lignin content were 44% and 44.9 % for Nalbari Source & Biswanath Chariali Source coir dust respectively.

Table 1: Proximate chemical analysis of the coir dust

<i>Particulars</i>	<i>Biswanath Chariali source (%)</i>	<i>Nalbari source (%)</i>
Ash Content	13.89	13.97
Calorific Value	Washed: 3820, Unwashed: 3850	Washed: 3825, Unwashed: 3857
Moisture Content	24.9	25.3
Solubility in Cold Water	10.5	11.2
Solubility in Hot Water	13.9	14.3
Solubility in 1% NaOH	17	17.2
Cellulose Content	36.56	35.90
Lignin Content	44.9	44
pH	6.64	6.65

4.2 Physical properties of Coir dust:

The results for physical properties of coir dust were shown in Table 2 & Table 3. The study shows the bulk density, porosity, swelling % (Table 2) and distribution of particles size of coir dust (Table 3)

Table: 2

Particulars	Biswanath Chariali source	Nalbari source
Bulk Density (Kg/ cm³)	156.393	155.202
Total Porosity (%)	74.4	75.75
Aeration Porosity (%)	18.8	20.05
Water Holding Capacity (%)	55.6	55.7
Swelling %	65.88	-

Table: 3 Determination of percent weight of graded Coir dust

Average particle Size (mm)	Weight of coir dust (gm)	Weight %
+10	30	3.0
-10 to +5.6	72	7.2
-5.6 to +4.75	70	7.0
-4.75 to 2.36	110	11.0
-2.36 to +1.18	196	19.6
-1.18 to +0.600	236	23.6
-0.600 to +0.300	222	22.2
-0.300	64	6.4



Figure 1- Particle size distribution of coir sample

4.3 Determination of viscosity of crude oil:

The oil sample is placed into a glass capillary U-tube and the sample is drawn through the tube using suction until it reaches the start position indicated on the tube's side. The suction is then released, allowing the sample to flow back through the tube under gravity. The narrow capillary section of the tube controls the oil's flow rate; more viscous grades of oil take longer time to flow than thinner grades of oil.

Table: 4

Rheology of raw crude	Sample 1			Sample 2			Sample3		
Temp °C	18	21	24	21	24	27	24	27	30
PV (C_p)	30	29	20	24	20	10	22	18	12
YV (dynes/cm²)	20	12	8	10	8	2	8	6	2

4.4 Determination of Sorption Capacity of graded coir dust in water & crude oil:

The sorption capacity of coir dust (graded) was checked both in water and crude oil. We made two samples of all graded coir dust; one was raw and another was heat treated at relatively low temperature. On heat treatment, the dried coir dust was kept in hot air oven at 150 °C for 17 minutes. The water sorption capacity decreases in heated coir dust due to melting of lypophylic extractive compounds in the pores of coir dust. The lypophylic extractive compounds like wax and gum melted and made hydrophobic thin film on the surface of the pore. Heat treatment also reduces the weight of the coir dust. The weight reducing of heated coir dust might be caused of the lack of water content and volatile extractive content. The results of the sorption of water and crude oil were shown in the table 5 and 6 respectively. Also graded coir dust vs. weights gained by coir dust after sorption of water and oil graphs were shown.

Table: 5 Weight gained by coir dust after absorption of water

Average particle Size (mm) of coir	Weight of Untreated coir dust sample after absorption of water (gm/gm of coir dust)	Weight of Treated coir dust sample after absorption of water (gm/gm of coir dust)
Sample (as received)	5.1564	4.5372
10	2.6039	2.0802
-10 to +5.6	3.0299	2.7555
-5.6 to +4.75	3.1505	3.0869
-4.75 to +2.36	3.8968	3.5293
-2.36 to +1.18	5.1148	4.8952
-1.18 to +0.600	5.9814	5.1521
-0.600 to +0.300	7.0810	6.6627

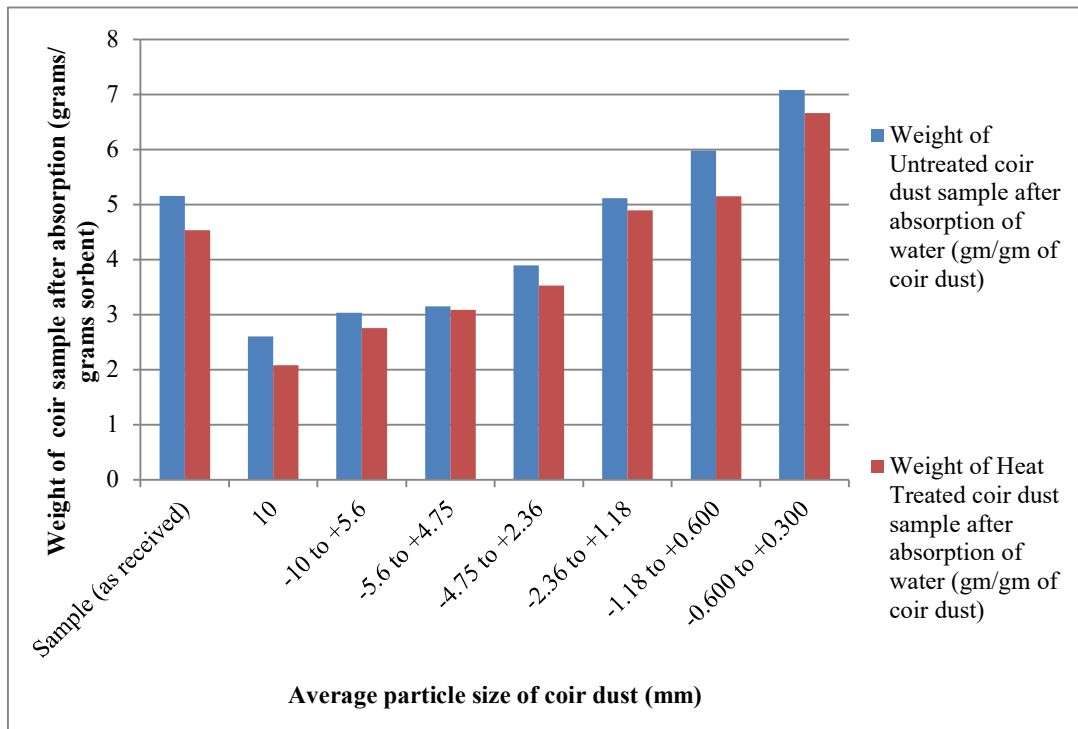


Figure 2- Absorption test of coir dust in Water

From the sorption test it was seen that water sorption was little decrease in heat treated coir dust. The above chart shows the change in absorption of coir dust in different particle size.

Table: 6 Weight gained by coir dust (CD) after absorption of crude oil

Average particle Size (mm)	Weight of Untreated CD sample after absorption of crude oil (gm/gm of CD)	Weight of Treated CD sample after absorption of crude oil (gm/gm of CD)
Sample (as received)	4.1772	4.2219
+10	1.5808	1.6184
-10 to +5.6	1.8124	1.8668
-5.6 to +4.75	2.3712	2.9084
-4.75 to +2.36	2.8410	3.7829
-2.36 to +1.18	3.5098	3.8019
-1.18 to +0.600	5.0289	5.8314
-0.600 to +0.300	5.9702	6.4438

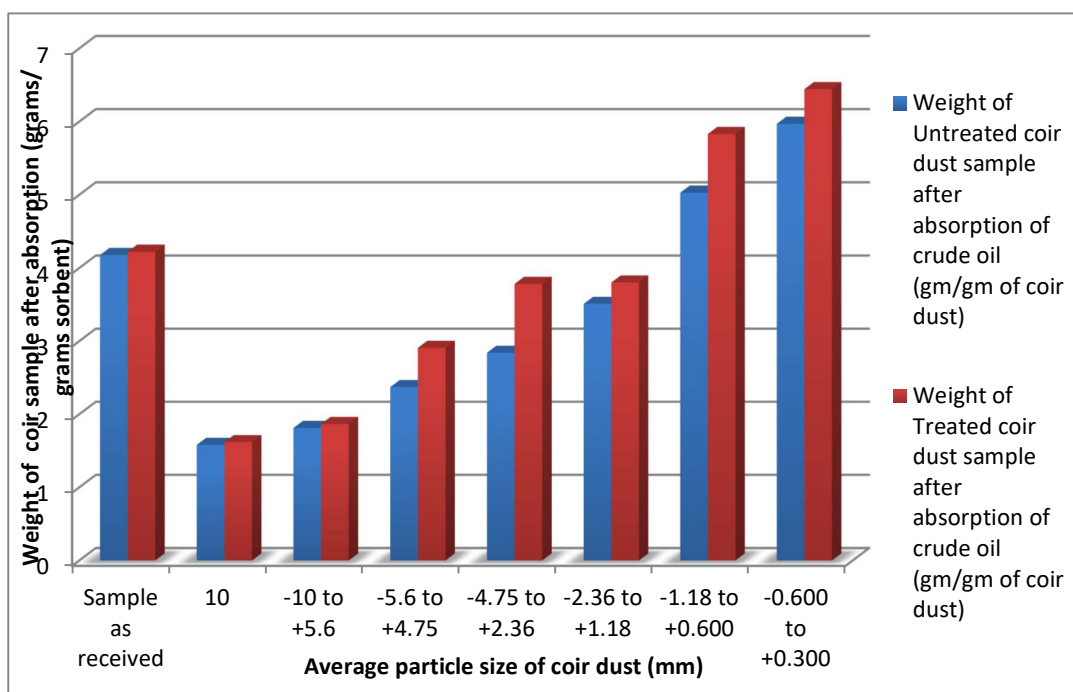


Figure 3- Absorption test of coir dust in crude oil

From these tests it was seen that in heat treatment there was increase in sorption of crude oil. It indicates that the hydrophobic character of coir dust increase since the lingo-cellulosic O-H bond reduces. From the above charts it was seen that absorption rate depends upon the particle sizes of the coir dust.

4.5 Sorption of water by the saw dust and mixture of saw dust and coir dust sample:

The saw dust have been heated at 70 °C for 30 minutes.

Table: 7

<i>Sample</i>	<i>Weight of the sample before absorption in gm</i>	<i>Weight of the sample after absorption in gm</i>
Saw dust	5.0055	31.01581
Coir + saw dust (1:1)	5 (2.5+2.5)	35.3377

4.6 Physico-Chemical Treatment of coir dust:

The Physico-chemical treatments were done to improve hydrophobicity and oil sorption capacity of coir dust. Hydrophobicity of coir dust was approached by using water sorption capacity of coir dust. Effects of various treatments onto coir dust were showed in Table 8 and Figure 4. Fig. 4 shows that the decrease of water sorption capacity on

Heated coir dust might be caused by melting of lypophylic-extractive compounds in the pores of coir dust. The lypophylic-extractive compounds, like wax and gum, melted and made a hydrophobic thin film on the surface of the pore. After heating at 150 °C for 17 minutes, the weight of heated coir dust decreased down. The weight reducing of heated coir dust might be caused of the lack of water content and volatile extractive content.

In Acetylation treatment, the decrease of water sorption capacity on acetylated coir dust related to the efficiency of acetylation process. The O-H groups of lignocellulose was reduced so the probability to form hydrogen bonding with water molecules decreased.

In acid treatment, the absorption of water and oil both increases simultaneously and in alkali treatment (Digestion treatment), it is seen that hydrophobic as well as oleophilic character improves and it absorbed 4.0869 g of water/ g sorbent and 8.0607 g crude oil/ g of sorbent. But in alkali treated (digested) coir dust the weight loss of coir dust is more as compared to other treatments.

Table: 8 Absorption of water and C. Oil of treated Coir Dust

<i>Coir dust (CD) Sample</i>	<i>Weight absorbed by CD by water absorption (gm/gm of sorbent)</i>	<i>Weight Absorbed by CD by crude oil absorption (gm/gm of sorbent)</i>
Raw	5.9814	5.0289
Heat treated	5.1521	5.8314
Acid treated	6.0750	6.6673
Acetylated	4.4339	6.7777
Alkali treated (Digested)	4.0869	8.0607

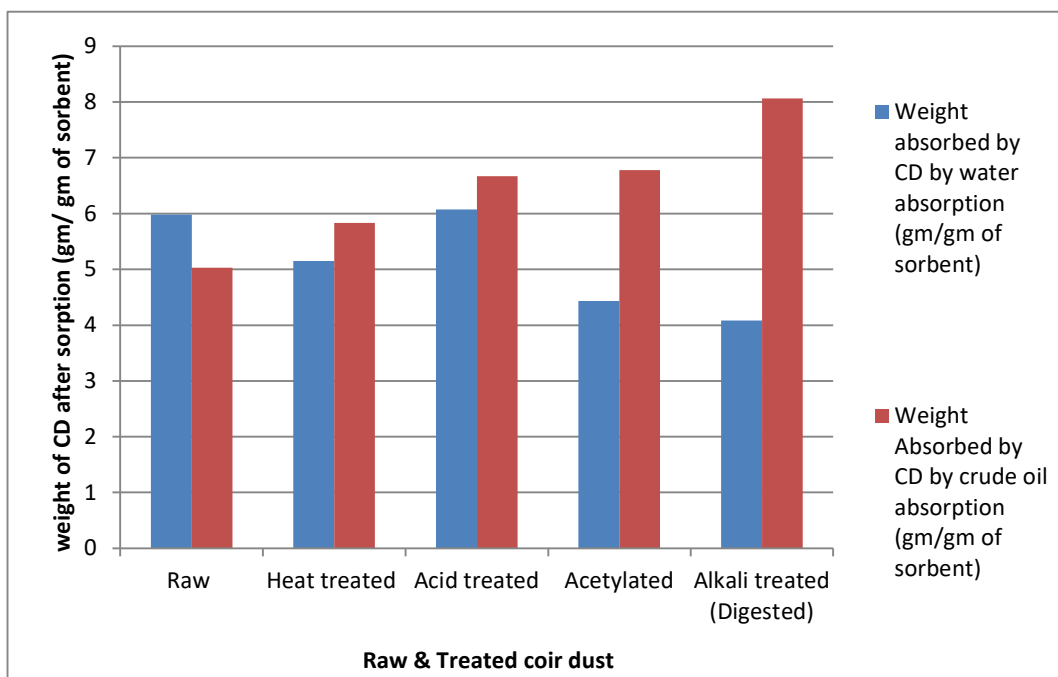


Figure 4 - Sorption test of raw and treated coir dust in water and C. oil

Esterified coconut coir dust was successfully done using oleoyl chloride by the replacement of hydroxyl groups of polymer backbone with that of acyl groups so as to achieve sufficient hydrophobic behaviour to interact highly with the similarly hydrophobic oil molecules.^[46] It was seen that with increase in concentration of oleoyl chloride from 10 % to 20 % and 30 % during esterification, the hydrophobic character of the formed coir dust-oleate was increased and also the oil sorption capacity was increased. When tested for oil adsorption studies, the oleoyl esterified coir dust (30%) was found to have better oil adsorption than the corresponding 10%, 20% oleoyl esterified coir dust and unmodified coir dust. The adsorption of crude oil was shown in Table 9 and figure 5. Based on the calculations, it was seen that the coir-oleate can serve as an alternative for oil spill due to its easy modification, abundance and biodegradability.

Table 9: Sorption of crude oil by raw and esterified coir dusts.

<i>Sample/Absorption</i>	<i>Weight absorbed by the coir dust after absorption of crude oil (gm/gm of sorbent)</i>
Raw coir dust	6.0310
Acylated coir dust (Esterified 10% Fatty Acid chloride)	9.6316
Acylated coir dust (Esterified 20% Fatty Acid chloride)	10.4612
Acylated coir dust (Esterified 30% Fatty Acid chloride)	11.3230

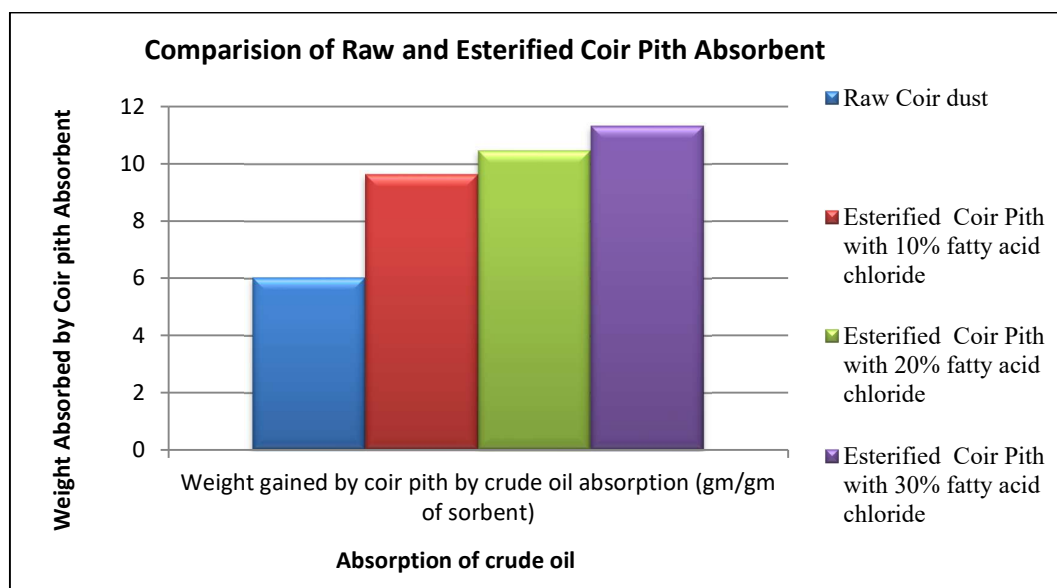
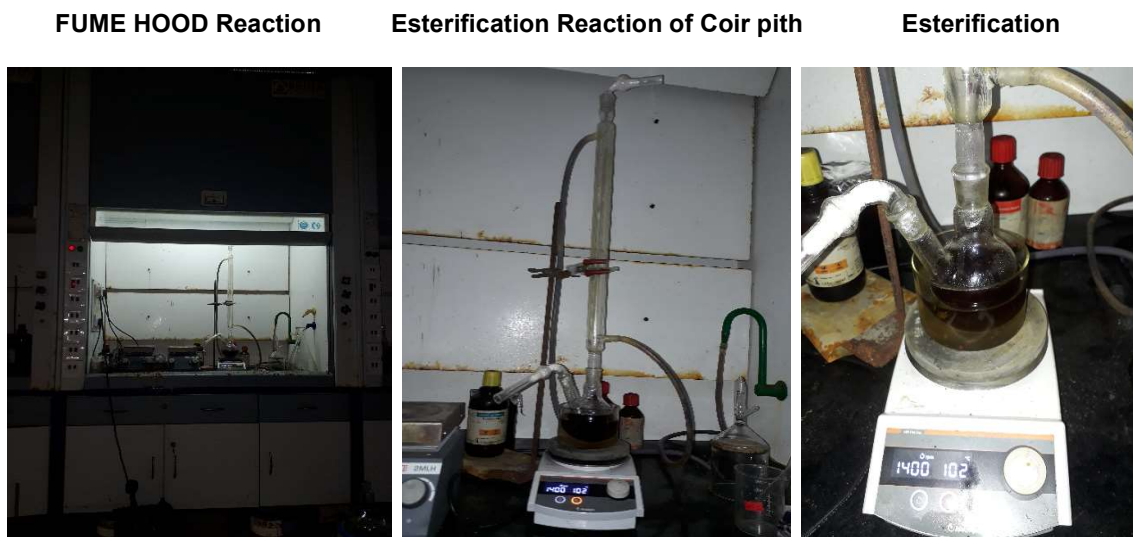


Figure 5 – Sorption of raw and esterified coir dust

Experimental Photos of Esterified Coir Dust



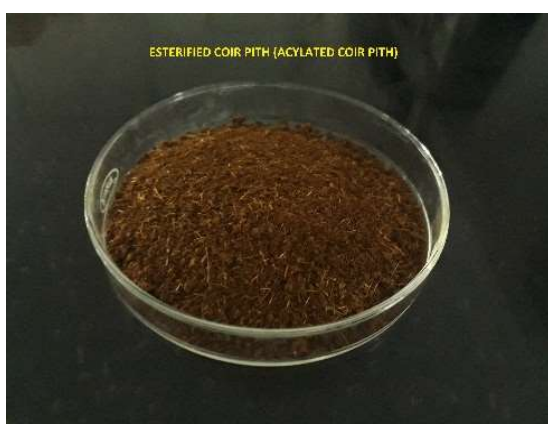
At the time of washing the Acylated Coir pith



Pre-treated Coir Pith (Boiled & Washed)



Esterified (Acylated) Coir Pith



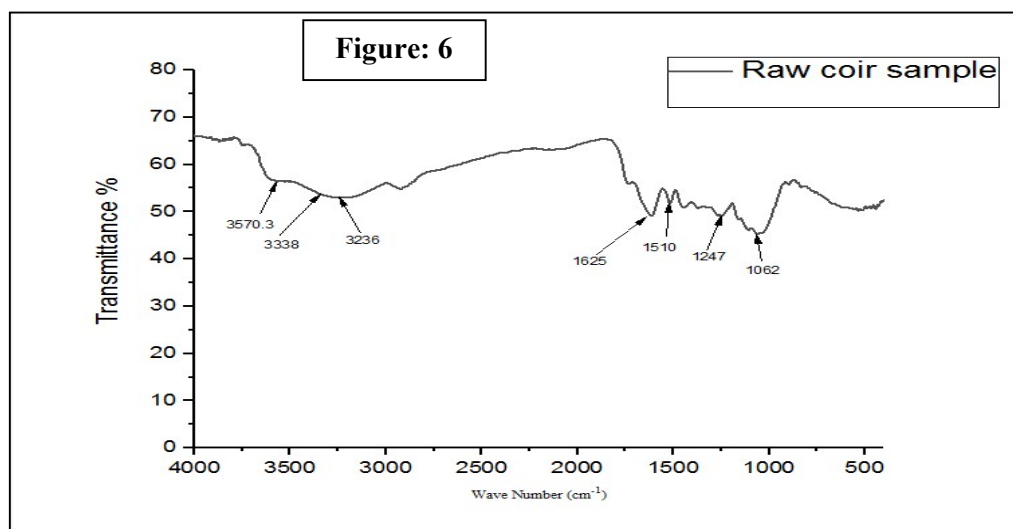
4.7 FT-IR spectra of Raw, Heat treated and Acetylated coir dust:

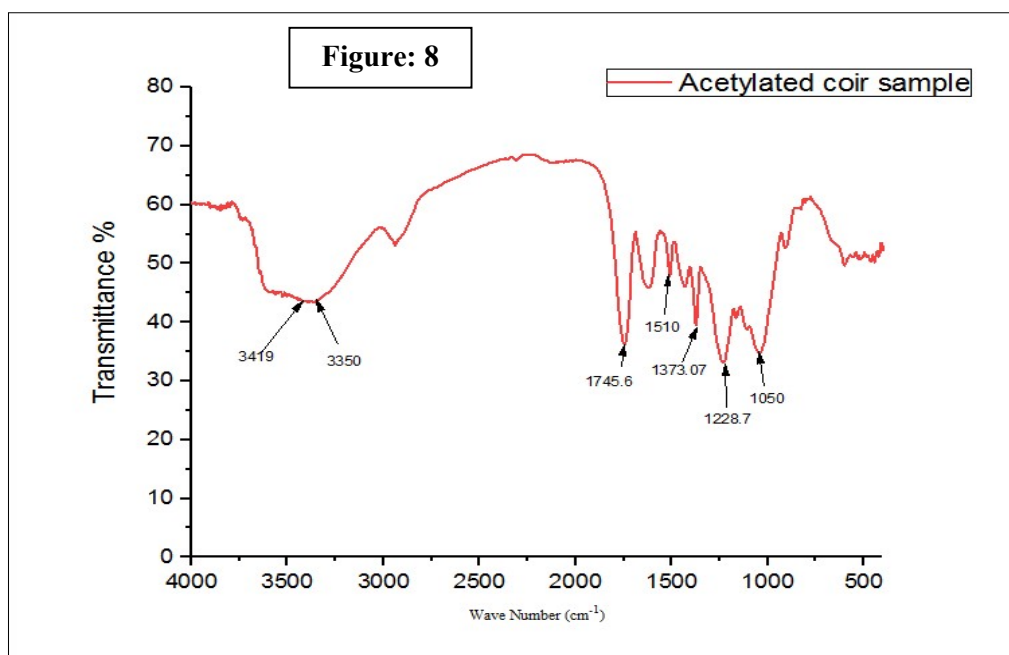
FT-IR spectra of the natural coir dust and the heated coir dust were showed in Figure 6 and Figure 7. Figure 6 and Figure 7 indicated no significant differences spectrum bands between unmodified coir dust and heated coir dust. The result indicated that heating treatment at 150 °C for 17 minutes didn't alter chemical structure on lignocelluloses of coir dust, only decreased the intensity of the O-H group absorption band at 3200-3300 cm⁻¹ in figure 7.

The FT-IR results indicate that coir dust has undergone modification by means of acetylation reaction (figure 8). The strong and sharp bands at 1745 cm^{-1} was attributed to absorption by C=O ester stretching, and at 1373.07 cm^{-1} was assigned to C-H stretching in -C-CH₃ group. Another strong band at 1228.7 cm^{-1} showed the C-O stretching in the acetyl group.

The decrease in the intensity of the O-H group absorption bands at 3419 cm^{-1} in spectrum of acetylation (figure 8) indicated that the acetylation was occurred to the O-H groups. The absence of absorption band at 1840 – 1760 cm^{-1} in spectrum of acetylation indicated that the acetylated coir dust was free of the unreacted acetic anhydride. The lack of absorption band at 1700 cm^{-1} stated that the acetylated coir dust was free of the byproduct of acetic acid also.

The other peaks 1510 cm^{-1} is for lignin & 1240 cm^{-1} for hemicelluloses.





4.8 Testing of Board making with the mixture of coir dust and binder:

Mechanical & Oil Sorption of Coir dust mixture with binder was shown in Table 10.



Figure 9- Coir dust Board of Nalbari & Biswanath Chariali Source of coir dust

Table 10: Mechanical and Physical Properties of composite Boards:

Sample	Density (g/cm ³)	Moisture content (%)	UTS (MPa)	MOR (MPa)	Breaking Load in tensile test (N)
Coir pith + LDPE	0.421	29.50	6.32 ± 0.12	12.51	885
Coir pith + Starch	0.445	33.40	4.16 ± 0.13	9.78	640
Coir pith + Starch (High Pressure)	0.681	28.10	4.86 ± 0.11	10.15	725
Coir pith + Foaming Agent	0.381	44.30	3.09 ± 0.14	6.35	620

Absorption of coir composite board in crude oil:

(i) Absorption of Coir block (Binder 1) in crude oil:

Make the coir pith block using binder 1 (LDPE). Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has

density **0.421 g/cm³** and weight of that piece was **12.7882 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 11 Weight gained by coir composite board after sorption of crude oil.

Time (in min)	15	30	45	75	105	135	235	335	435	565	3 days
Weight absorbe d by the coir block	17.337	18.571	19.183	20.145	21.115	22.273	25.023	26.860	27.923	28.077	30.168

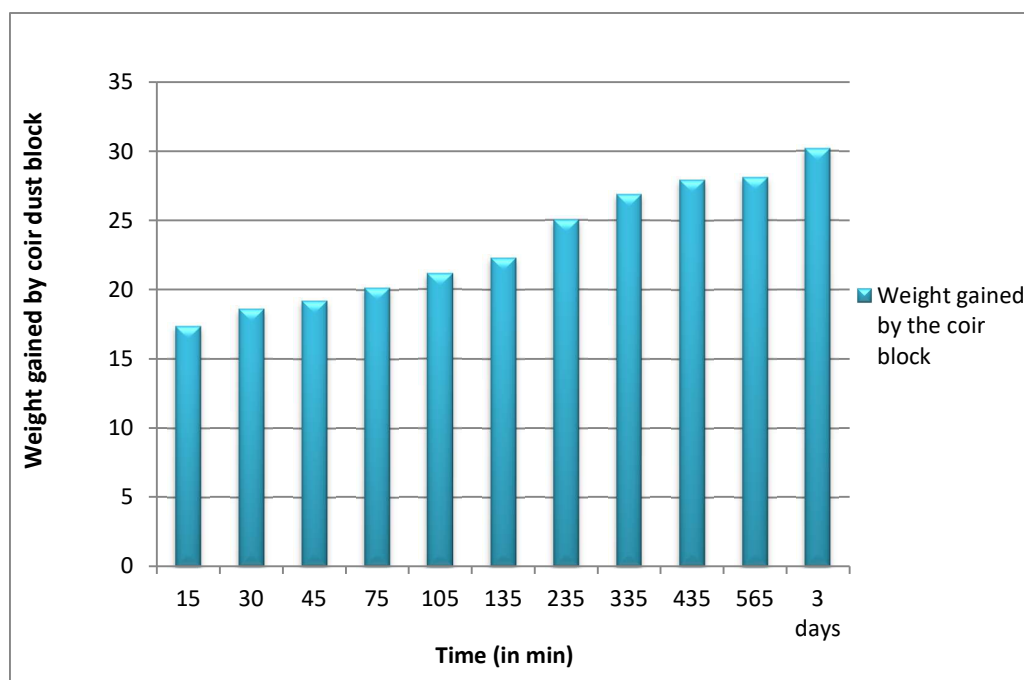


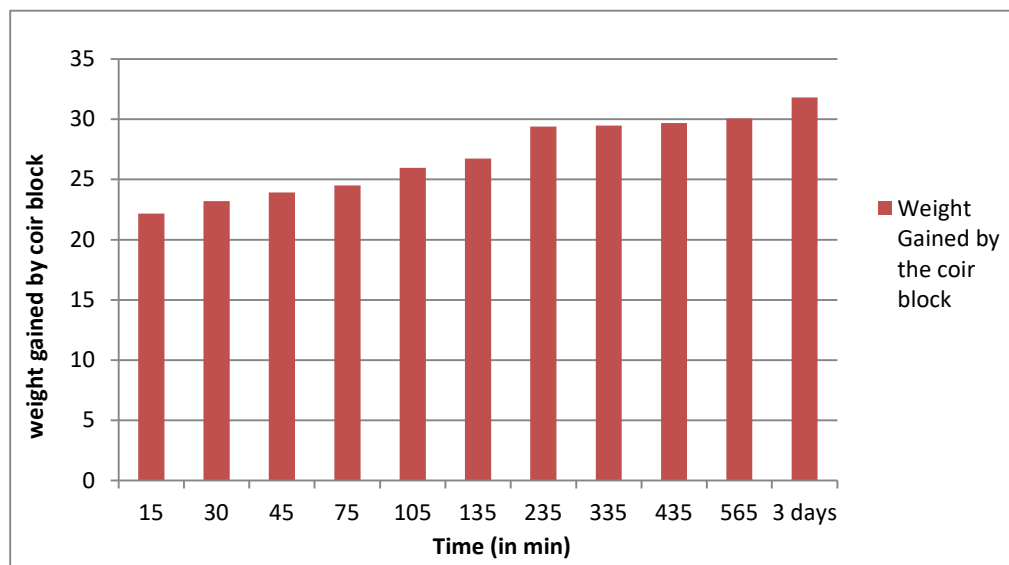
Figure 10: Absorption Rate vs. Time graph

(ii) Absorption of Coir block (Binder 2) in crude oil:

Make the coir pith block using binder 2 (Starch). Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.352 g/cm³** and weight of that piece was **14.9267 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 12 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	45	75	105	135	235	335	435	565	3 days
<i>Weight absorbed by the coir block</i>	22.166	23.200	23.901	24.494	25.971	26.734	29.394	29.451	29.682	30.052	31.801

**Figure 11: Absorption Rate vs. Time graph****(iii) Absorption of Coir block (Binder 2, High pressure) in crude oil:**

Make the coir pith block using binder 2 (Starch) with high pressure. Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.681 g/cm³** and weight of that piece was **13.7951 g**.

The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 13 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	45	75	105	135	235	335	435	565	3 days
<i>Weight absorbed by the coir block</i>	15.726	16.137	16.564	17.145	17.810	18.391	19.340	19.641	20.511	20.864	20.906

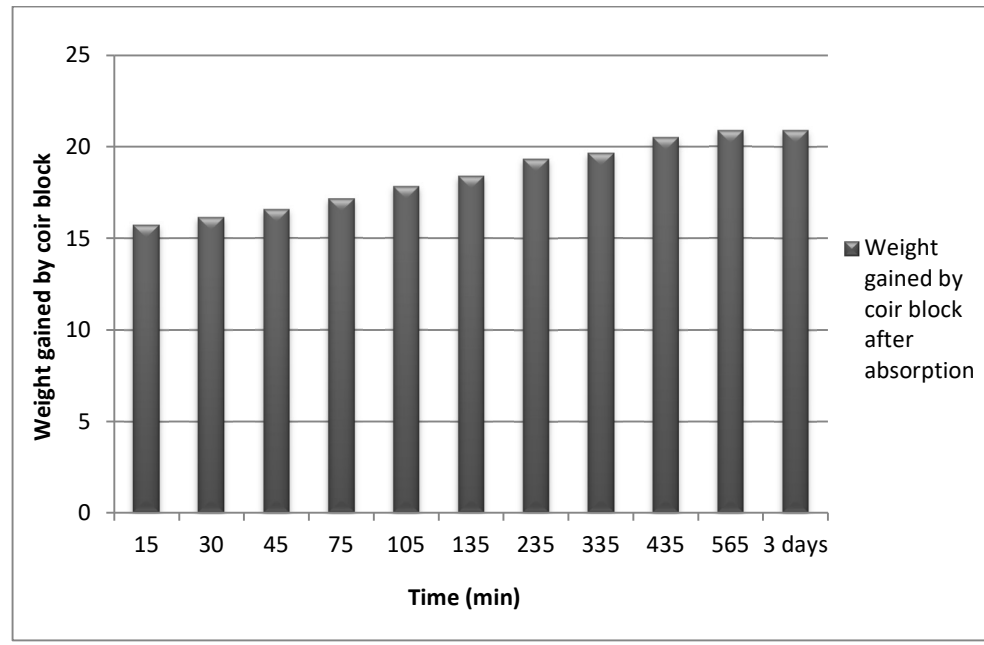


Figure 12: Absorption Rate vs. Time graph

(iv) Absorption of coir block (Binder 3, Biswanath Chariali source) in crude oil:

Make the coir pith block using binder 3 (Foaming Agent) to Biswanath Chariali source of Coir pith. Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.311 g/cm³** and weight of that piece was **14.9328 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 14 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	60	90	150	210	300	1 Day
<i>Weight absorbed by the coir block</i>	40.971	40.974	41.224	41.817	42.354	43.062	43.771	44.649

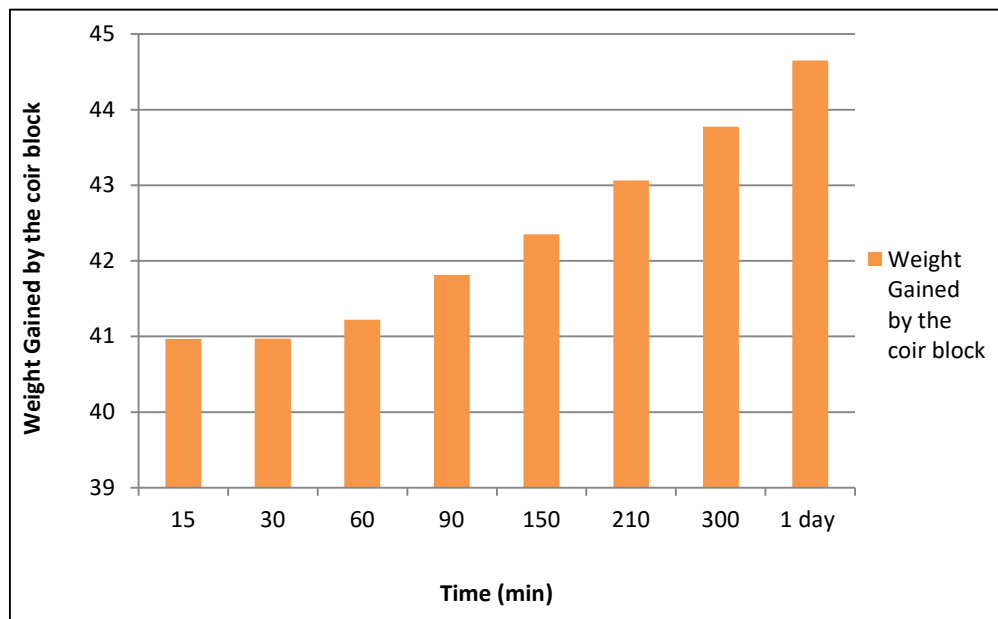


Figure 13: Absorption Rate vs. Time graph

(v) Absorption of coir block (Binder 3, Nalbari source) in crude oil:

Make the coir pith block using binder 3 (Foaming Agent) to Nalbari source of Coir pith. Cut the blocks into small pieces for perform absorption test in crude oil. Take one piece of block which has density **0.381 g/cm³** and weight of that piece was **16.6160 g**. The piece was then put in crude oil (50 ml) and checks its absorption rate with respect to times.

Table: 15 Weight gained by coir composite board after sorption of crude oil.

<i>Time (in min)</i>	15	30	60	90	150	210	300	1 Day
<i>Weight absorbed by the coir block</i>	30.480	32.445	33.420	34.175	35.292	35.953	36.566	37.959

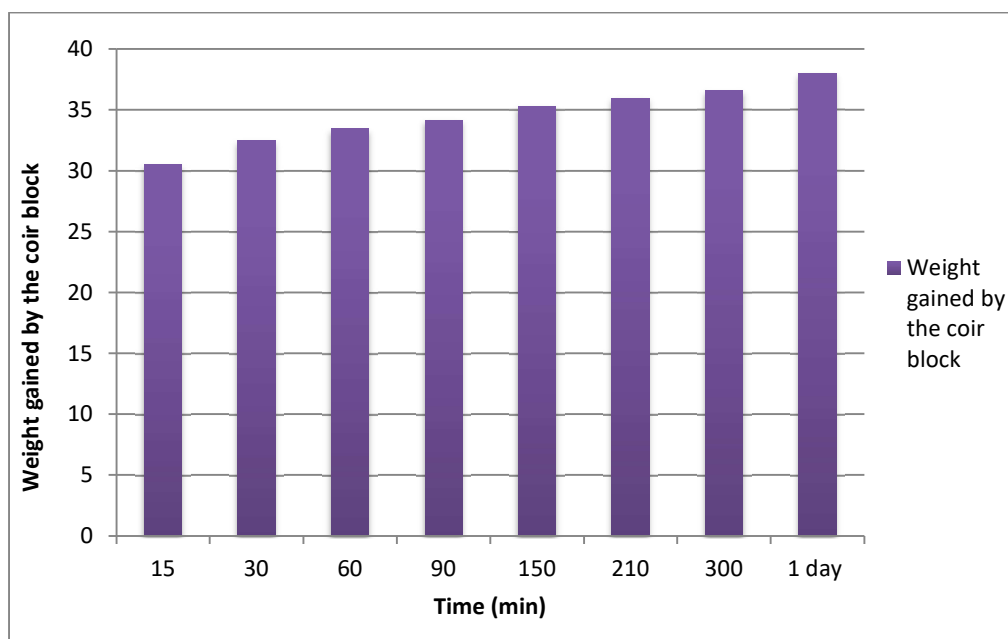
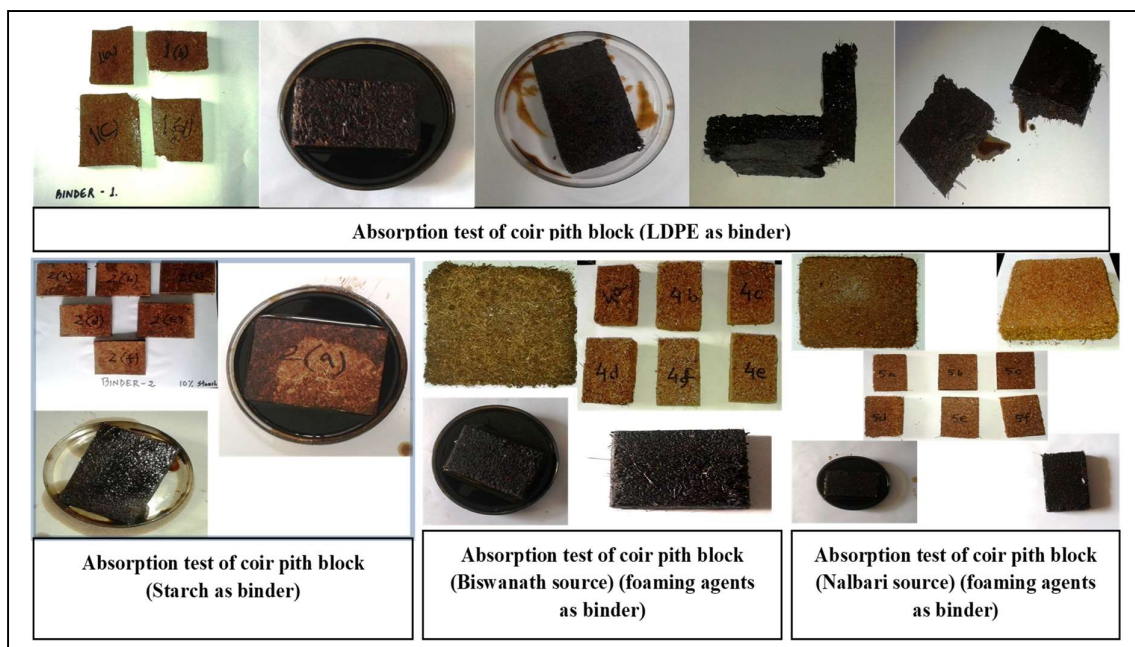


Figure 13: Absorption Rate vs. Time graph

The absorption rate of crude oil was better in coir pith block of Biswanath Chariali source (binder 3). It has fastest and highest absorptive capacity among all other coir pith block. Nalbari source of coir pith has better absorption rate with binder 1.

Experimental Photos



4.9 Testing of coir dust bags for absorption of crude oil:

To use coir practically for prevention of oil spillage in the oil fields and other affected areas we have prepared coir pith bags/ pouches. We make bags of markin cloth for containing treated coir dust and check its absorption rate in crude oil.

Weight of the coir dust bag we taken is 45.0861 g. In a beaker we prepare mixture of crude oil and water and mixed it by stirring. Then add the bag to it for 15-30 minute. After absorption, we take out the bag and weighed it.

Weight Gained by the coir dust bag is 107.9720 g.

Weight gain % is 70.54 approx.



4.10 Testing of coir dust handmade paper (round) for absorption in crude oil:

The machine we used for making coir pith and coir-cotton paper is beating machine, sheet form machine and screw press machine. Snaps of machines are given below.

Also the absorption in water & crude oil was shown below.



Beating Machine

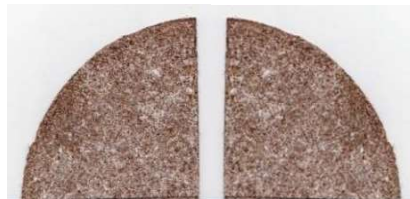


Sheet form Machine

Figure 14 – Beating Machine, Sheet form Machine, Screw press machine,



Screw Press Machine



Handmade coir pith paper

Table: 16 Absorption of water and crude oil by handmade paper.

<i>Coir-Cotton Paper/ Absorption</i>	<i>Weight Gain % by paper after Water Absorption</i>		<i>Weight Gain % by paper after Crude oil Absorption</i>	
	<i>20% cotton mixed coir paper</i>	<i>10% cotton mixed coir paper</i>	<i>20% cotton mixed coir paper</i>	<i>10% cotton mixed coir paper</i>
Untreated coir-cotton paper	465.30	494.93	389.86	458.71
Heat treated coir-cotton paper	384.78	371.10	358.78	434.72

4.11 Testing of coir dust of various treated handmade paper for absorption test in crude oil and water :

The absorption rate of **Oven dried** handmade coir pith paper, **Hot plate press** handmade coir pith paper, **Oil treats** handmade coir pith paper, **Latea treats** handmade coir pith papers are checked for both crude oil and water. The weight gain% of each paper after absorption was shown in below:

Table: 17. Variously treated coir dust paper/ sheet exposed to water & C. oil

<i>Treated Coir-Cotton Paper/ Absorption</i>	<i>Oven Dried handmade coir pith paper</i>	<i>Hot Plate press handmade coir pith paper</i>	<i>Oil treated handmade coir pith paper</i>	<i>Latea treated handmade coir pith paper</i>
<i>Weight Gain % by paper after Water Absorption</i>	487.84	446.30	140.20	343.17
<i>Weight Gain % by paper after Crude oil Absorption</i>	370.90	384.16	86.06	266.80

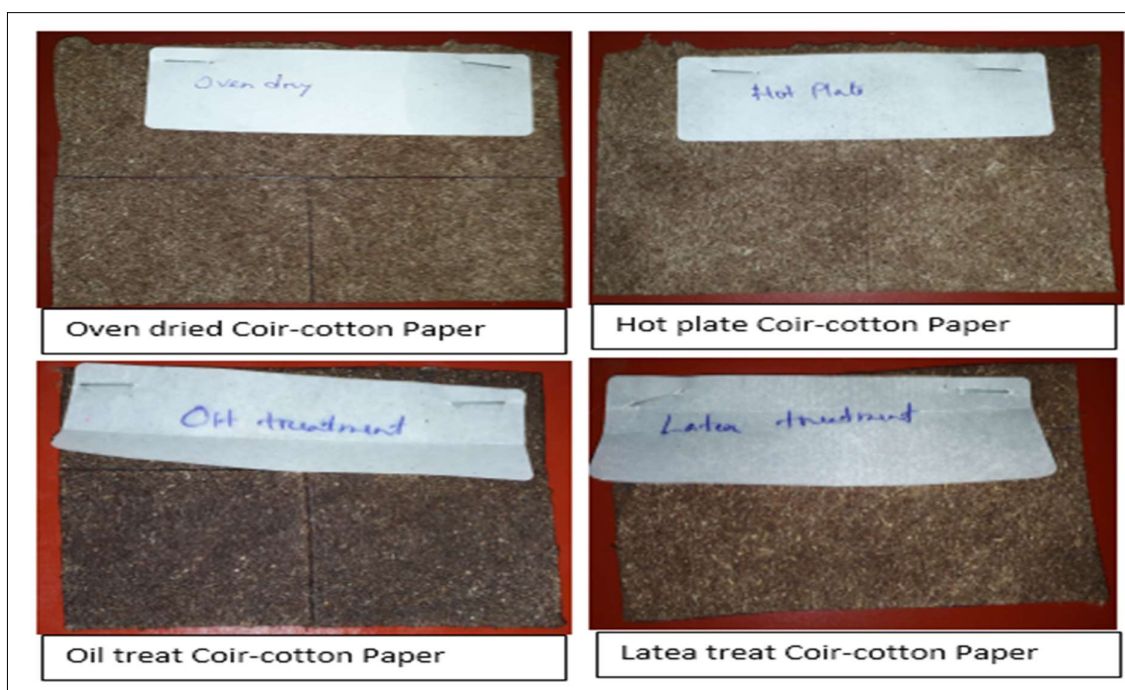


Figure 15- Various treated coir-cotton Paper

4.12 Evaluation of their Physico-chemical properties of Coir Dust, Jute Waste, Cotton Waste :

Physico-chemical properties of CD, JW and CW are represented in Table18. The thickness of cotton waste and jute waste were measured as 0.32 and 1.43 mm and the densities of coir dust, cotton waste and jute waste recorded 0.759 ± 0.2 , 1.55 ± 0.5 and 1.45 ± 0.5 g/mm³. The cellulose content of coir dust, cotton and jute waste were determined and found 35.50 ± 2.0 , 94.50 ± 3.5 and 60.23 ± 4.0 % while lignin content recorded 44.20 ± 3.0 , 15.4 ± 2.5 and 13.3 ± 2 % in cotton and jute waste respectively.

Table 18: Physico- chemical properties of coir dust, cotton and jute waste

Sample	Thickness (mm)	Moisture Content (%)	Ash Content (%)	pH	Density (g/cm ³)	Lignin (%)	Cellulose (%)
Coir dust	-	80.00 ± 5.0	3.40 ± 0.1	5.30 ± 0.5	0.759 ± 0.2	44.20 ± 3.0	35.50 ± 2.0
Cotton waste	0.32 ± 0.05	7.50 ± 0.5	1.75 ± 0.05	7.25 ± 0.6	1.55 ± 0.5	15.4 ± 2.5	94.50 ± 3.5
Jute waste	1.43 ± 0.5	9.93 ± 0.8	0.68 ± 0.05	6.74 ± 0.5	1.45 ± 0.5	13.3 ± 2	60.23 ± 4.0

4.13 FTIR studies of CD, JW, CW and their Composite Mat (OAM):

The representative FTIR spectra of natural waste fibres, coir dust and composite mat (OAM) were shown in Fig. 16. The FTIR spectra of CW fibre and JW fibre [Fig. 16 (a) & (b)] showed a broad and intense band at $\sim 3420\text{ cm}^{-1}$ due to the hydrogen bonded O-H stretching vibration from the cellulose. The IR band at $\sim 2931\text{ cm}^{-1}$ for JW fibre is assigned to $-\text{CH}_2$ anti-symmetric stretching vibration in cellulose and degraded hemicelluloses and lignin. This band is absent for cotton fibre, which may be due to absence of residual lignin resulting in decrease carbon atoms attached to carbon or hydrogen ($-\text{C}-\text{C}-$ or $-\text{C}-\text{H}$) [26]. Band at $\sim 1630\text{ cm}^{-1}$ in both CW and JW fibre is assigned for bending form of absorbed water and due to some involvement of carboxylate group. The $1050\text{--}1250\text{ cm}^{-1}$ region bands involve the C-O stretching vibrations of aliphatic primary and secondary alcohols in cellulose. Both the fibres show a peak at $\sim 889\text{ cm}^{-1}$ due to β -glycosidic linkage of glucose ring of cellulose indicating the typical structure of cellulose [27, 28]. FT-IR spectra of coir dust show characteristics peaks for $-\text{C}=\text{O}$ stretching of the carbonyl and acetyl group in the 4-O-methyl-glucuronoacetyl xylan component of hemicelluloses in coir fibre $\sim 1702\text{ cm}^{-1}$. The band at $\sim 1521\text{ cm}^{-1}$ for coir dust is due to presence of aromatic rings of lignin. A band at $\sim 1247\text{ cm}^{-1}$ was observed for untreated coir fibre which may be attributed to $-\text{C}-\text{O}-\text{C}-$ bond in the cellulosic chain [28]. In the composite board, the individual characteristics FTIR peak of cotton/jute and coir appears with slight change in position and intensity indicating cross linking of coir and natural fibres.

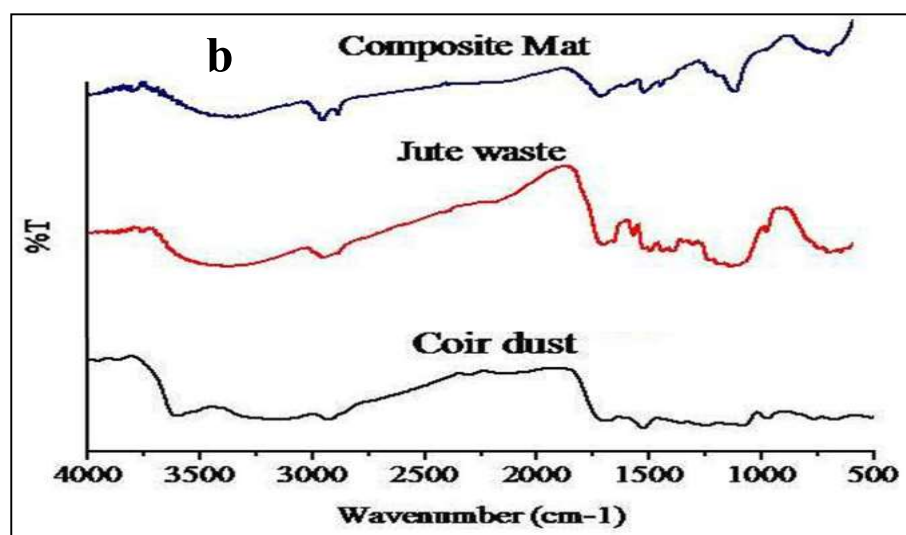
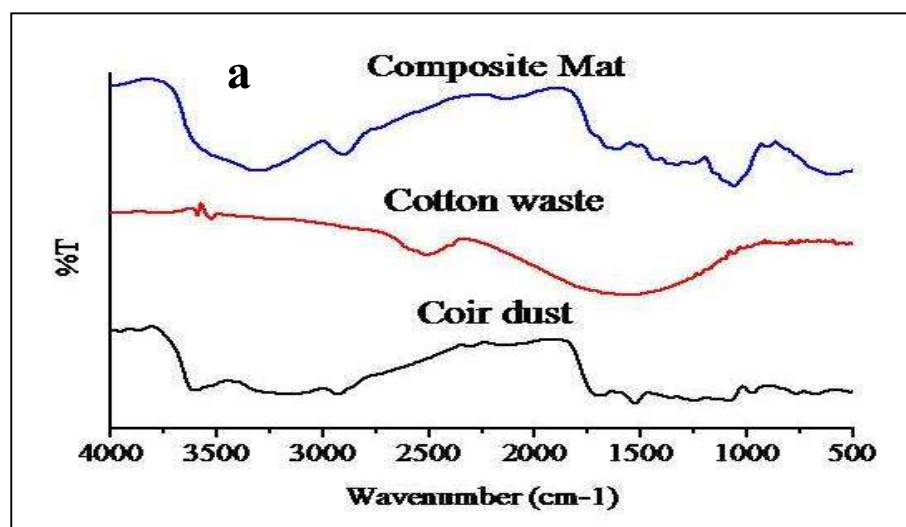


Figure: 16 FT-IR spectra of (a) Cotton waste, coir dust and CD+CW composite mat, (b) Jute waste, coir dust and CD+JW composite mat.

4.14 XRD studies of CD, JW, CW and their Composite Mat (OAM):

The XRD studies of natural waste fibres, coir dust and composite mat (OAM) were carried out to investigate the crystallinity of the samples at different stages and were shown in Fig. 17 (a) & (b). The XRD patterns of CW fibre and JW fibre [Fig. 17 (a) and (b)] showed two peaks representing the planes 101 and 002 at angles 2θ which are subtended at 12.8° and 19.8° respectively, characteristic of cellulose crystalline phase of the fibre [27, 28]. Crystallinity Index (CI) was calculated and it

is found to be 45 % and 58 % for waste JW fibre and CW fibre respectively. This higher value of crystallinity index for CW fibre is consistent with higher value of cellulose with insignificant quantity of lignin. In the XRD of coir dust, an extensive broadening peak with 2θ value in the range of 15.6° and 26.8° are observed due to the characteristic diffraction peak of 101 and 002 planes [29, 30].

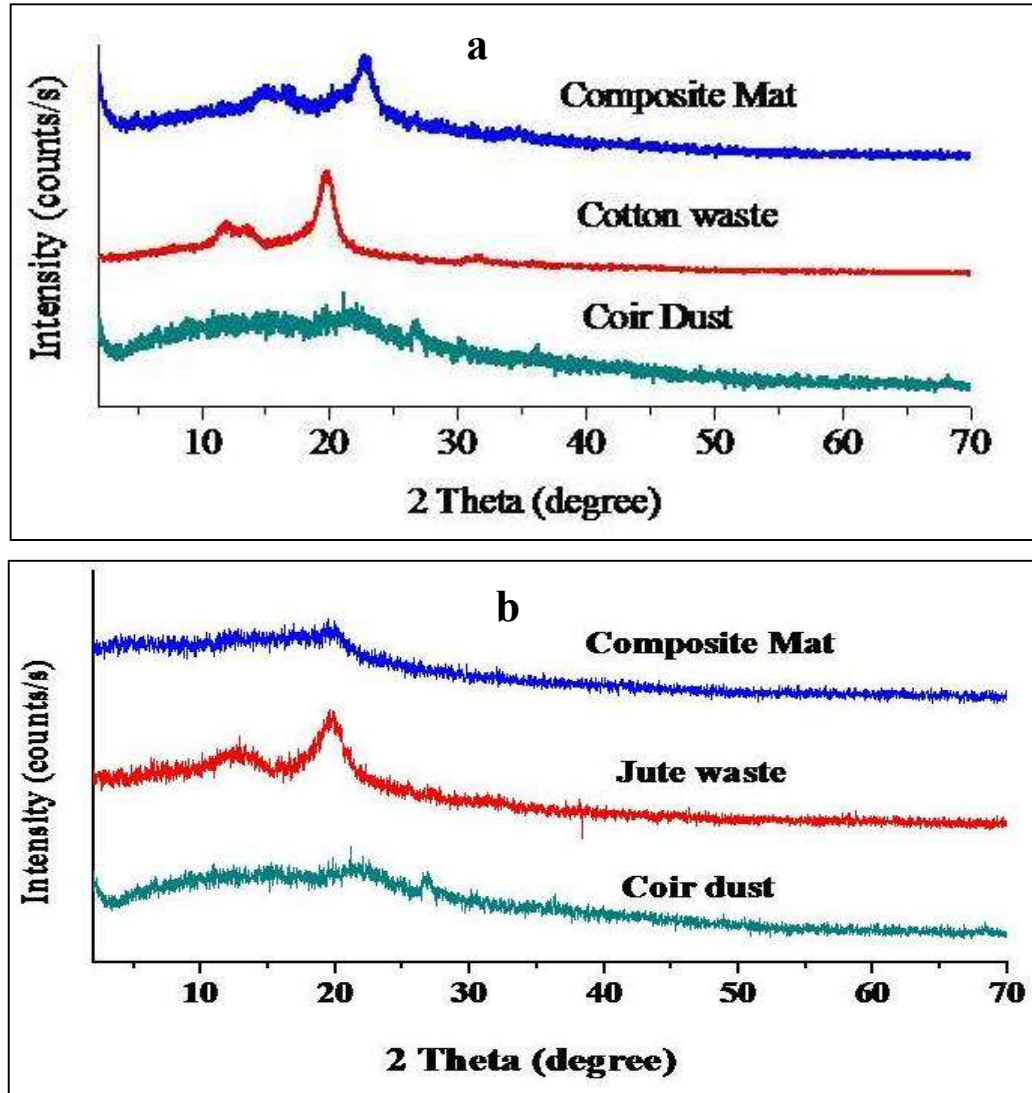


Figure 17: X-ray diffraction spectra of (a) Cotton waste, coir dust and CD+CW composite mat, (b) Jute waste, coir dust and CD+JW composite mat.

The XRD of OAM shows slightly boarder and weaker reflection compared to those of coir and waste fibres which may be attributed to the significant structural modification in the OAM. This modification in comparative intensity and location of reflection peak indicates a three dimensional linkage-formation between coir dust and natural fibre during composite formation.

4.15 TGA studies of CD, JW, CW and their Composite Mat (OAM):

The TGA curves of natural waste fibres, coir dust and composite mat (OAM) were represent in **Fig. 18 (a) & (b)**. The different weight loss percentage obtained from thermogravimetry analysis for particular samples are represented in **Table 18**. The weight loss in the samples occurred in three stages: the first one in the range of 20-120 °C, the second one 120-400 °C and third one 400-650 °C. For the waste fibres, weight loss in the range of 20-120 °C are attributed to the evaporation of absorbed and crystal water molecule associated with the cellulose fibre [31]. The actual degradation occurred in the range of 120-400 °C are assigned for disintegration of polymeric materials such as hemicelluloses, α -cellulose, lignin etc. and the third stage (400-650 °C) is attributed to the carbonization of these polymeric materials [32]. Similarly for coir dust, the first one (7.90 %) refer to the loss of water molecules along with some volatile oily components present in the coir fibre (20-120 °C); second (49.04 %), because of the thermal degradations of polymeric materials (120-400 °C); and the third stage (10.85 %) in the range of 400-650 °C is attributed to carbonization of these polymeric materials [33-35]. It is observed that OAM exhibited weight loss of 56.90 % and 66.49 % for JW mat and CW mat respectively in the transition temperature 120-400 °C compared to 57.81 % and 80.29 % weight loss for jute fibre and cotton fibre individually. Similarly in the transition temperature 400-650 °C, JW composite and CW

composite exhibit weight loss 10.41 % and 10.23 % respectively compared to individual fibres, which was in the range 10.85-19.60 %. This shows that the cellulose of waste fibres and cellulosic coir dust interact strongly forming a three dimensional network and thereby increased the thermal stability of the respective OAM.

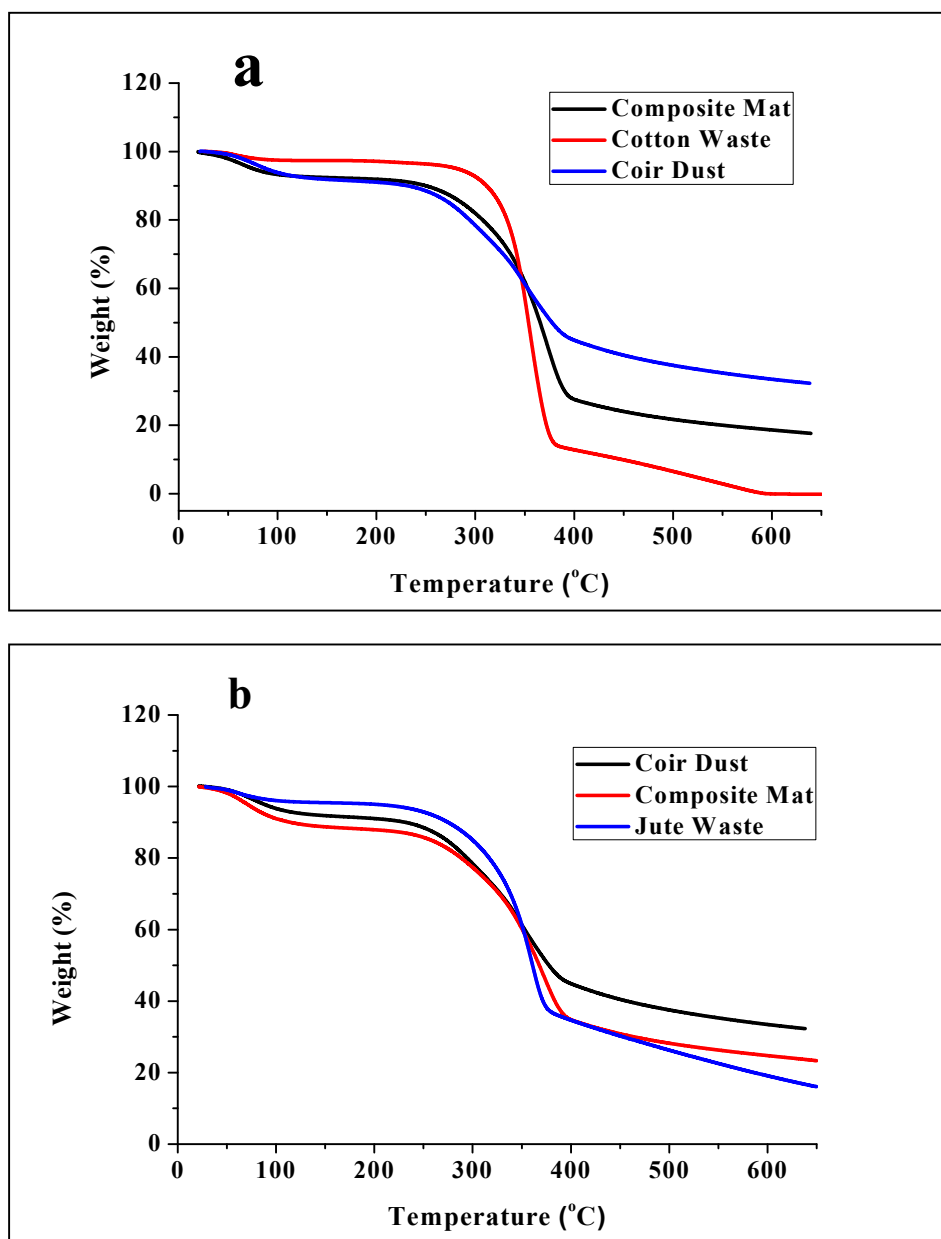


Figure 18 : TGA spectra of (a) Cotton waste, coir dust and CD+CW composite mat, (b) Jute waste, coir dust and CD+JW composite mat.

Table 18: % Weight loss of different samples in various stages

Samples	% weight loss		
	20-120 °C	120-400 °C	400-650 °C
Coir Dust	7.90	49.04	10.85
Jute waste	7.41	57.81	19.60
Cotton waste	5.60	80.29	11.56
Composite Board (CD +JW)	8.98	56.90	10.41
Composite Board (CD +CW)	6.23	66.49	10.23

4.16 Microstructural characteristics (SEM) of CD, JW, CW and their composite

OAM:

Fig. 19 (a-e) & 20 (a-e) represent the SEM and invert SEM of CD, CW, JW and OAM. The SEM of CD [Fig. 19 a (i) & (ii) and 20 a (i) & (ii)] shows that fibre surface was covered with oils, waxes and extractives layer which is the part of natural constituent of fibres(Das et al. 2014). It also observed from the SEM of coir dust that very rough surface and shallow pits were also visible of coir dust [36]. Fig. 19 (b) & 20 (b) represented the SEM of cotton waste fibre and observed that the fibres are long, continuous, thin and whitish in colour. Also observed that, the fibres are forming a tough network without adding of any binder as well as no pores or longitudinal cracks are perceptible on the surface. Fig. 19 (c) & 20 (c) represents the SEM of JW fibre. Jute fibres are continuous, uniform and cylindrical in shape

which is comparable with CW fibre. Some bundle forms of fibre are also observed [37]. The SEM of the composite mat prepared from cotton waste and coir dust are presented in Fig. 19 (d) & 20 (d). A strong network of cotton and coir fibres consistently bonded by guar gum solution has been seen. This similar network of two natural fibres may be due to superior fibrillation taken place during stock preparation in beating. Fig. 19 (e) & 20 (e) shows the SEM of composite mat made from coir dust and JW. In jute fibres, comparatively larger in diameter, fibres are clearly visible. The coir fibres are fixed with the jute fibres uniformly forming a strong network of jute and coir fibres. During stock preparation, the void spaces between coir and jute fibre are occupied by guar gum. Due to some void spaces between coir and natural fibres, oil absorbency may also increased. The gum solution used in beating operation during stock preparation helps to enhance the cohesiveness of both the fibres. Although, natural fibres have similar morphology, but there are differences among them which may be because of the deviation of morphological properties like number of fibre cells, cell wall size, size and shape of the lumen etc. Therefore, different fibre plants show different characteristics as well as mechanical properties [38]. Figure 21 (a-f) represent the surface plot diagrams of coir dust, cotton waste, jute waste and composite mat made from mixture of coir dust and cotton waste or jute waste, which reflect their surface morphologies. Some rough stimulus were seen randomly throughout in the micrographs of coir dust, cotton waste and jute waste, [Fig. 21 (a), (b), (c) & (d)], whereas they are low distinguishable as well as with relatively smoother surface in the micrographs of absorbent mat which is made from the mixture of CD and CW or JW [Fig. 21 (e) & (f)].

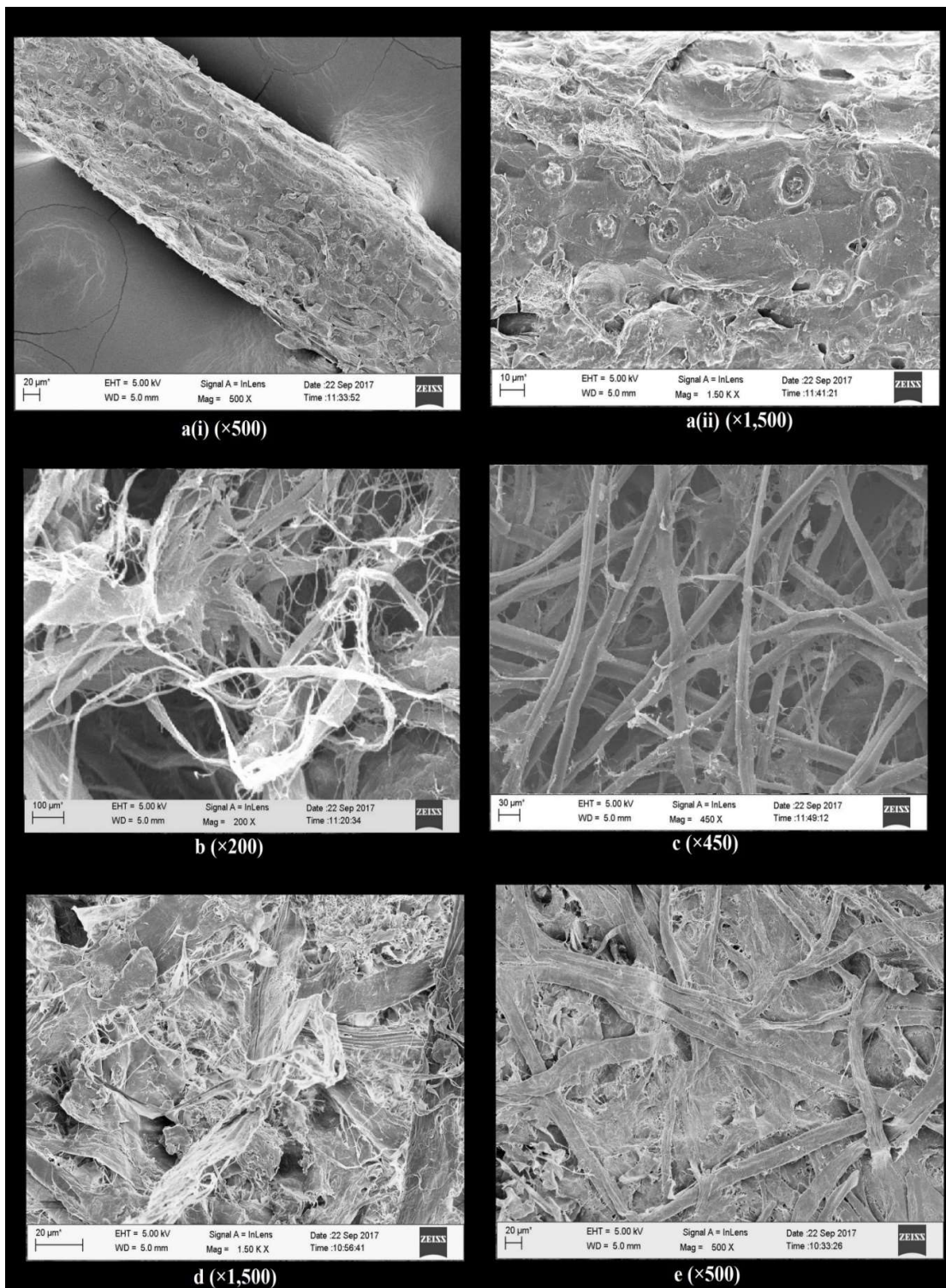


Figure 19: SEM of (a) Coir Dust, (b) Cotton waste fibre, (c) Jute waste fibre, (d) Composite made from Coir dust + cotton waste, (e) Composite made from Coir dust + jute waste.

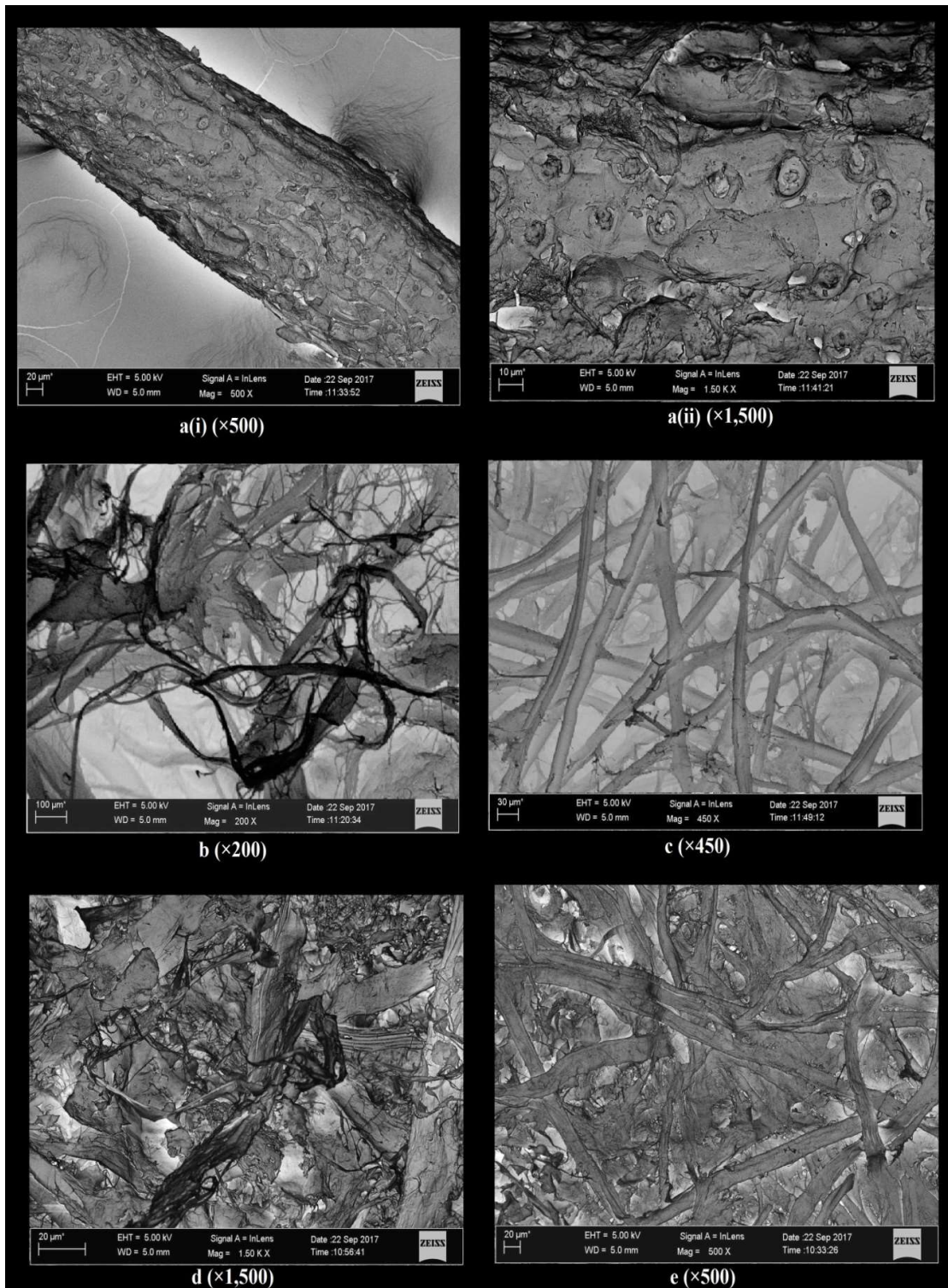


Figure 20: Invert SEM of (a) Coir Dust, (b) Cotton waste fibre, (c) Jute waste fibre, (d) Composite made from CD + CW, (e) Composite made from CD+JW.

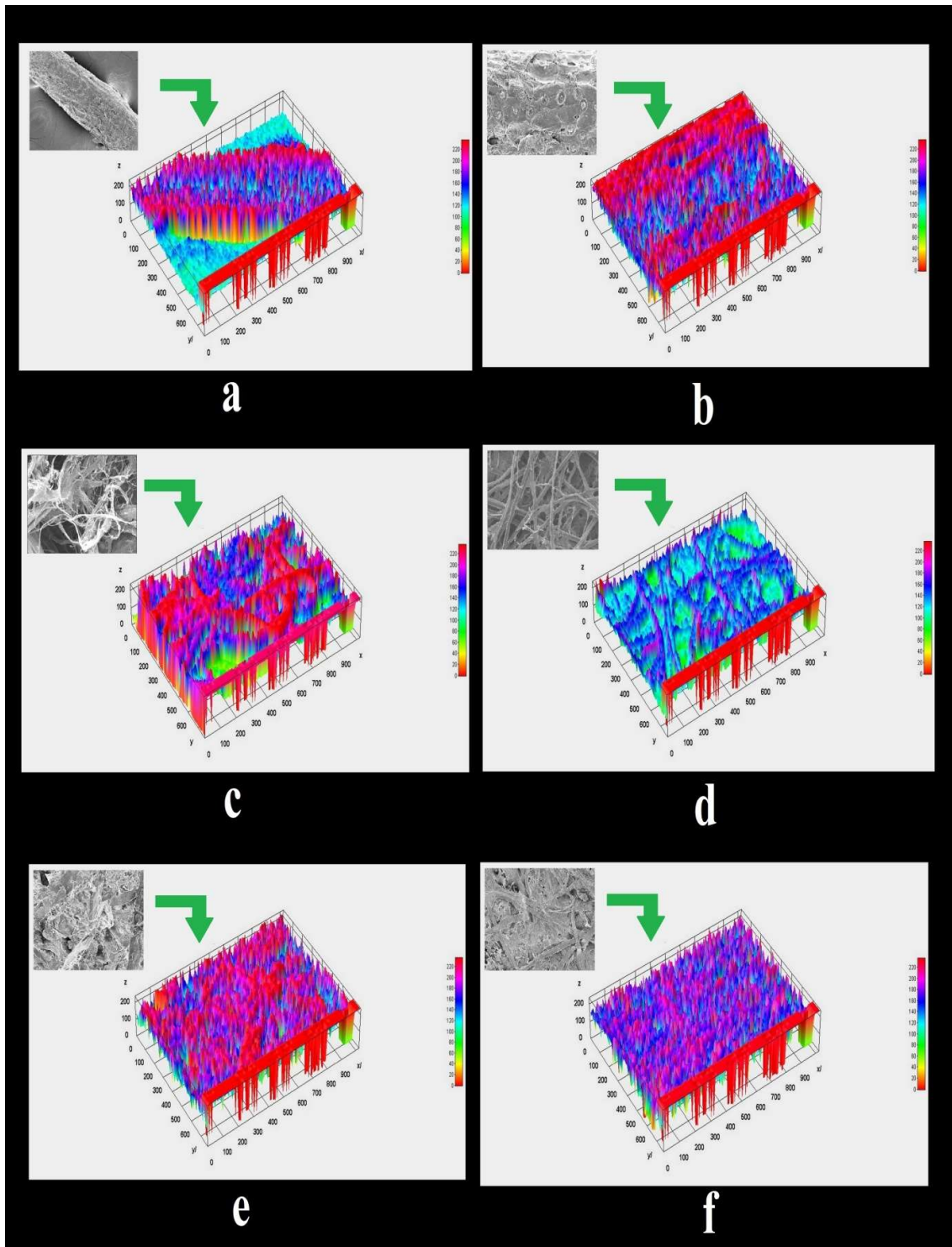


Figure 21: Surface SEM analysis of (a) Coir Dust, (b) Cotton waste fibre, (c) Jute waste fibre, (d) Composite made from CD + CW, (e) Composite made from CD+JW.

4.17 Mechanical Properties of JW, CW and their Composite OAM:

The mechanical properties like ultimate tensile strength (UTS), elongation of CW and JW are represented in **Table 19**. The UTS of JW and CW were recorded as 250.62 ± 6.0 and 235.00 ± 5.0 MPa respectively which are shown in the **Fig. 22**. The superior tensile strength may attribute to presence of cellulose substance in the natural fibre [37]. Rowell studied that lower cellulose content don't contribute much towards the tensile strength [39]. Likewise, similar elongation values were also recorded for jute waste and cotton waste which may be stated as 12.35 ± 2.5 and 11.65 ± 2.0 % respectively. The mechanical or physical strength properties of natural fibre materials were found with similar results without showing much deviation which is compatible for making composite mats.

Table 20 represents the mechanical properties of the OAM prepared from the mixture of coir dust and cotton waste or jute waste. Similar densities were recorded which 0.258 ± 0.03 and 0.249 ± 0.06 g /cm³ for the mats are made from the mixture of CD & CW and CD & JW respectively in the ratio of 85:15. Different densities were recorded in mats with differing ratios i.e. 90:10, 85:15 and 80:20 may be due to the gradual increment of natural fibre [37]. The ultimate tensile strength properties of OAM prepared in combination of CD and CW or JW at different stock ratios have also been shown in **Table 20**. It has been observed in the **Fig. 23**, the OAM manufactured from CD & CW showed comparatively lower tensile strength (43.86 ± 3.5 MPa) compare to that of mats made from CD & JW (49.17 ± 4.0 MPa). Natural fibre improved the mechanical strength properties, which is due to the uniform bonding between natural fibre and coir dust fibre [37]. Natural fibre forms a regular network with the coir fibre, that causes improved ultimate strength properties. Senthil also observed similar results along with increased tensile strength of the composite mats due to the presence of cellulose content in the natural fibre [40]. Rowell also observed the same result in the composite mats

prepared from the mixture of CD & CW/ JW [39]. A natural binder i.e. guar gum was used in the mixture sock, which integrates to improve the physical strength of the final absorbent mat product. **Table 20** represents certain mechanical properties like elongation, folding and bursting properties of the absorbent mats, where 85:15 ratio was found to be encouraging with superior results compared to other ratios. Hence, the above ratio of coir dusts and natural fibres has been found to be optimum to get high mechanical strength properties in the absorbent mats.

Table 19: Mechanical properties of jute and cotton waste fibre

<i>Sample</i>	<i>UTS (MPa)</i>	<i>Elongation (%)</i>	<i>Breaking Load in tensile test (N)</i>
<i>Jute waste</i>	250.62±6.0	12.35±2.5	3215±30
<i>Cotton waste</i>	235.00±5.0	11.65±2.0	2955±25

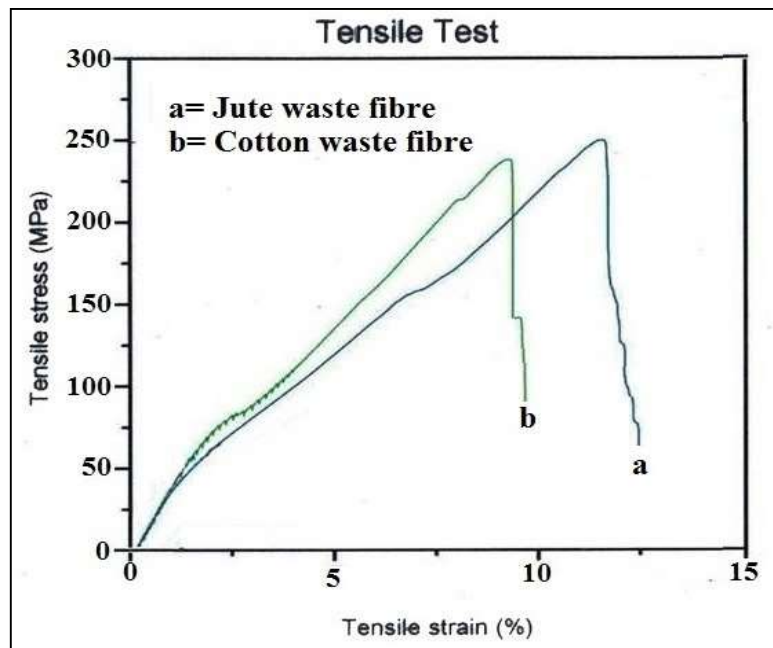


Figure 22: Tensile strength of jute waste fibre and cotton waste fibre.

Table 20: Mechanical properties of composite made from coir dust, cotton and jute waste:

Sample	Moisture content (%)	Density (g/cm³)	UTS (MPa)	Elongation (%)	Breaking Load in tensile test (N)	Folding endurance (DF/min)	Bursting strength (kg/cm²)
CD+ CW (90:10)	39.40±1.2	0.244±0.02	30.76±3.0	4.78±0.5	2340±15.0	440±5.0	5.60±1.5
CD+ CW (85:15)	36.10±1.0	0.258±0.03	43.86±3.5	18.15±1.5	2525±25.0	1446±15.0	8.80±2.0
CD+ CW (80:20)	32.80±0.8	0.281±0.02	29.42±3.0	4.35±0.8	2450±10.0	406±9.0	6.80±1.8
CD+ JW (90:10)	45.20±1.3	0.238±0.05	35.85±3.5	4.00±1.0	1940±10.0	98±4.0	3.30±0.5
CD+ JW (85:15)	42.00±1.2	0.249±0.06	49.17±4.0	23.86±2.0	2895±15.0	1850±20.0	9.70±1.0
CD+ JW (80:20)	38.20±1.1	0.258±0.05	38.38±3.0	17.08±1.5	2950±15.0	750±5.0	6.20±1.2

Coir Dust=CD, Cotton waste =CW, Jute waste =JW

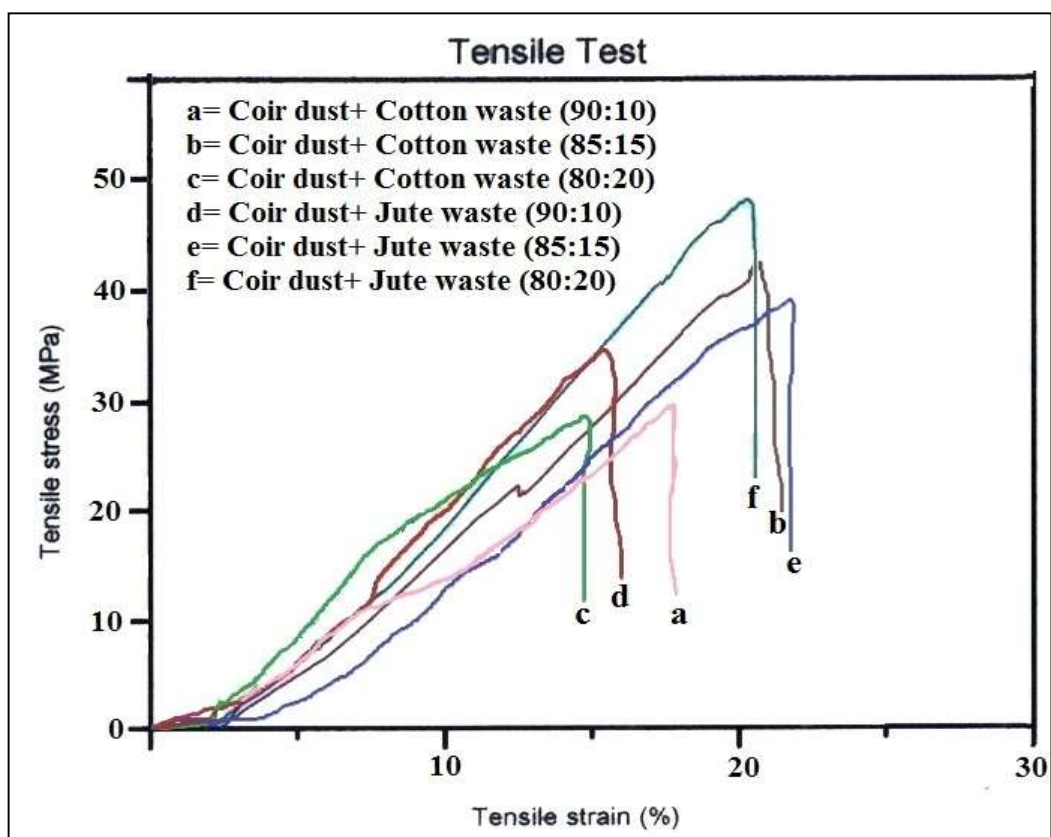


Figure 23: Tensile strength of composite made from Coir dust and Cotton waste (90:10).

a) Coir dust and Cotton waste (90:10), b) Coir dust and Cotton waste (85:15), c) Coir dust and Cotton waste (80:10), d) Coir dust and Jute waste (90:10), e) Coir dust and Jute waste (85:15), f) Coir dust and Jute waste (80:10).

4.18 Oil Absorption Properties of OAM:

Table 21 represents the oil absorption properties of the OAM prepared from the mixture of CD and CW or JW in different oils like crude oil, diesel oil, hydraulic oil, transformer oil, Mobil oil. Oil absorption values also show comparable & superior results in the mats made from the CD & CW and CD & JW in the ratio of 85:15 at 20-30 °C. Presence of natural fibre as well as more cellulose containing cotton fibre such as CD & CW composite mat may show better oil absorbent results than the composite made from CD & JW. It is evident that oil absorbency is better in low density oil than the high density ones.

Table 22 represents the comparison of various commercial or known oil absorbents model with OAM. Oil absorbency of OAM shows better performance than commercial or known oil absorbents model in different types of oil.

Table 21: Oil Absorption properties of composite made from coir dust, cotton waste and jute waste

Sample	Crude Oil (g/g)	Diesel Oil (g/g)	Hydraulic Oil (g/g)	Transformer Oil (g/g)	Mobil Oil (g/g)
After 20 min at 20-30 °C					
CD+ CW (90:10)	21.0±2.0	17.1±1.8	21.3±2.1	17.5±1.5	18.0±1.5
CD+ CW (85:15)	21.8±2.0	19.2±2.0	19.0±1.5	18.6±1.8	20.4±2.0
CD+ CW (80:20)	18.1±1.5	14.9±1.0	17.1±1.5	14.3±1.0	15.2±1.5
CD+ JW (90:10)	18.9±1.5	19.6±1.5	18.4±2.0	18.4±1.5	16.6±2.0
CD+ JW (85:15)	19.6±2.0	19.1±1.6	19.6±2.0	18.6±1.5	19.6±1.8
CD+ JW (80:20)	18.4±2.5	17.9±2.3	17.5±1.5	15.4±0.5	18.9±1.5

Oil Density:

**Crude Oil=880 kg/m³, Diesel Oil= 820 kg/m³, Hydraulic Oil= 874 kg/m³,
Transformer Oil= 800 kg/m³, Mobil Oil= 840 kg/m³**

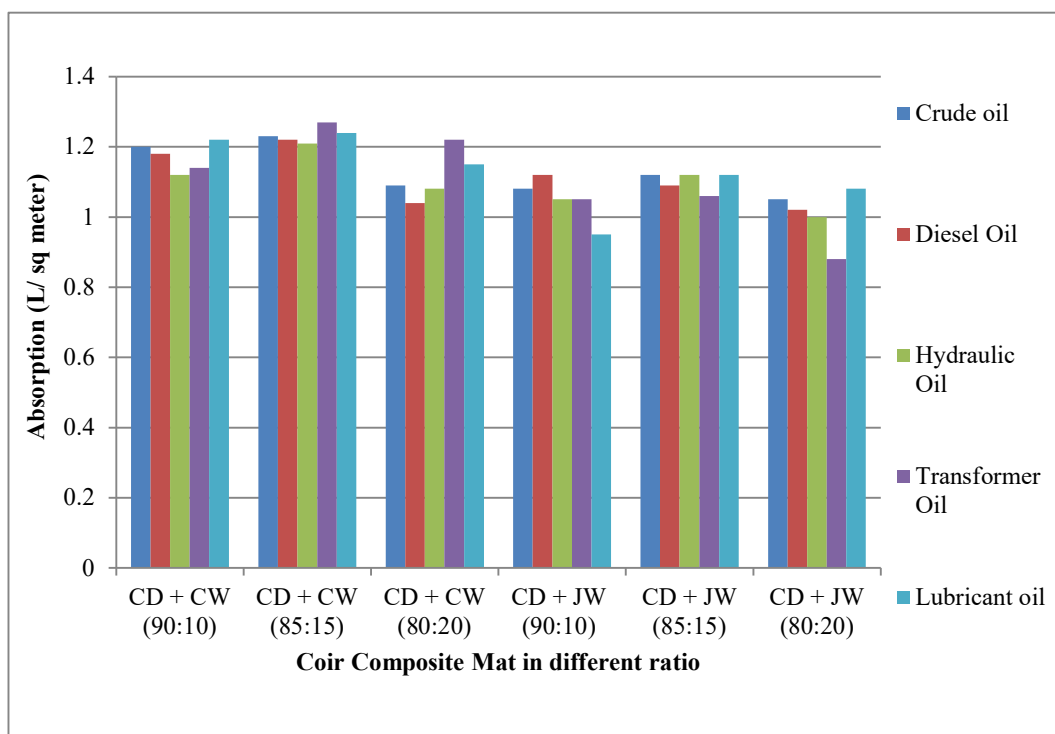


Figure 24: Absorption of various tested oil by Oil Absorbent Mat (OAM)

Table 22: Comparison of Various Oil Absorbents

Oil Absorbent Media	Oil	Oil Absorbency (g/g)	Reference
Oil absorbent mat (OAM) [CD+ CW (85:15)]	Crude	21.8±2.0	Present work
	Diesel	19.2±2.0	
	Hydraulic	19.0±1.5	
	Transformer	18.6±1.8	
	Mobil	20.4±2.0	
Swellable Porous PDMS	Diesel	12.0	41
	Crude Oil	9.0	
PDMS sponge	Transformer	4.3	42
Cotton towel	Diesel	5.0	43
Commercial PP nonwoven mat	Hydraulic	11.7	44
	Crude	8.1	

Polyvinyl-Alcohol Formaldehyde Sponges	Hydraulic	12.8	44
	Crude	12.9	

4.19 Recovery and Reusability:

Recovery and reusability is a significant factor for oil absorbent. In order to measure the possible for oil recovery from OAM, we used mechanical extraction approach. With the help of hydraulic press at 25 N of pressure, OAM were compressed while suspended over, until no further oil extruded from the compressed mat. The resultant oil was weighed immediately, and this process was repeated for 10 cycles. Interestingly, the absorption capacity of OAM started higher (19.8 ± 0.1 g/ g) and then capacity decreased (11.6 ± 0.2 g/g) slowly after 10 cycles. **Fig.25** represents the recycle oil absorption capacity of OAM (CD & CW; 85:15) in each cycle. After 10 cycles, the absorption capacity of OAM was retained at 60 % compared to the first cycle. But after 4 to 5 cycle cracks are observed on OAM and it goes on deterioration.

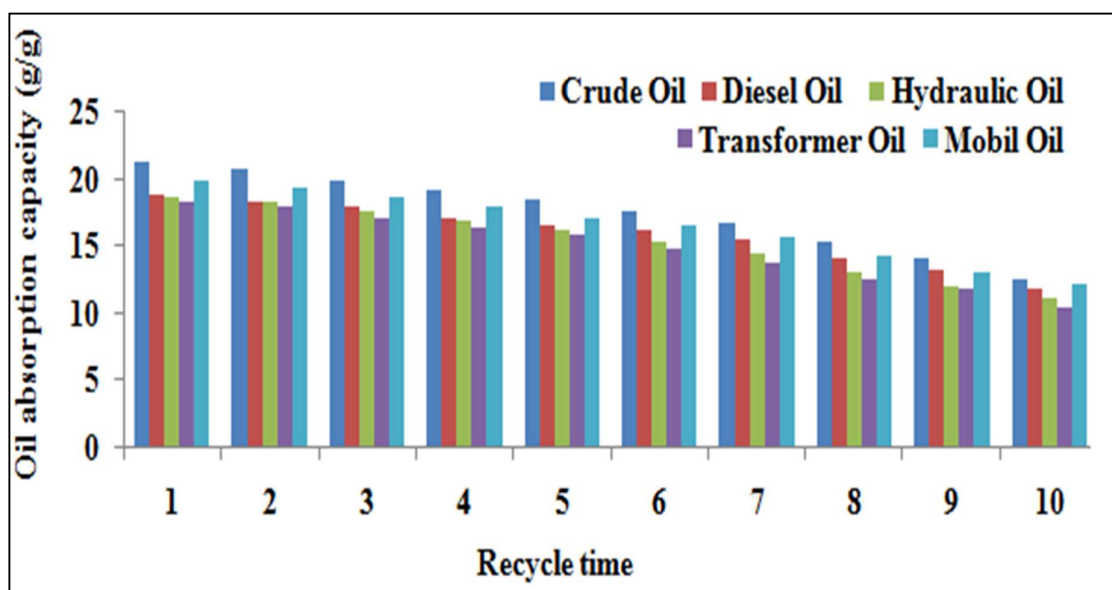


Fig. 25: Recycle oil absorption capacity of composite made from coir dust and cotton waste (85:15).

Chapter 6

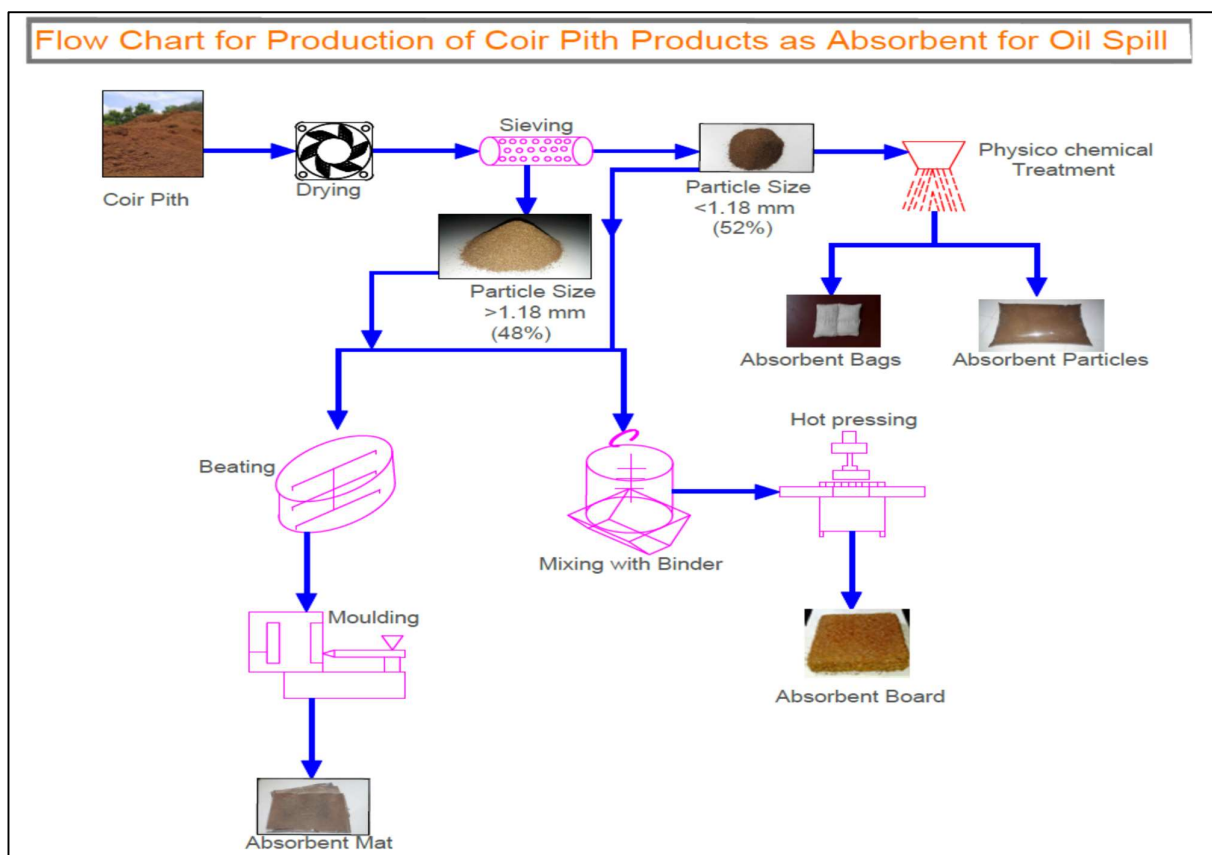
Conclusion

Natural organic sorbents, such as coir dust, are environmental-friendly, biodegradable and inexpensive materials if compared to polymer synthetic materials. Some of the natural organic sorbents have good sorption capacities, but they also sorb water as well (low hydrophobicity). This is a disadvantage when used in water environments. Hydrophobicity of the sorbent is an important parameter that determines the effectiveness of sorbent. Some physicochemical treatments were done to improve the hydrophobicity of coir dust such as heating, acetylation, alkali treatment or Acylation (Esterification), while in esterification, 30% Coir-oleate can serve as an alternative for oil spill due to its easy modification, abundance and biodegradability with more oleophilic character as compared to other.

From the present study it may be concluded that coir dust, a waste produced from the coir industry, can be converted to the form of pouches/bag, block, mat/pad by addition of natural fibre and also by adopting certain mechano-chemical progression. The mats or sheets prepared from blending of cotton wastes or jute wastes with coir dusts exhibited better oil absorbency, with adequate physical strength properties, and that makes them suitable to be used in case of oil spills in oil exploration, industries, transportation and in machine/vehicle maintenance works etc. As the mat/pad is biodegradable or recyclable in nature, it can be easily disposed to soil or environment after extracting the oil from the mat/pad. Recycling of such industrial wastes for making useful products not only helps the society, but also helps to conserve the ecosystem and environment.

COIR PITH PRODUCTS as ABSORBENT

Flow Chart for production of Coir pith Products as Absorbent for oil spill



Photographs of Developed Products preparation for field trial:



Figure 26: Developed products from coir dust

Patent Filed in India (Patent Application No. 0015NF2019):

A patent entitled “High Oil Absorbing Mat/ Pad” was filing jointly by CSIR NEIST and Coir Board.

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ANITA DAS RAVIDRANATH (COIR BOARD),
SUBODH CHANDRA KALITA (CSIR-NEIST),
PINAKI SENGUPTA (CSIR-NEIST).

Poster Presentations:

1. A poster was Presented in the **Materials Research Society of India (MRSI)**, North-East Chapter conference on The Frontier Chemical Biology, 26-28 June, 2018 jointly organised by CSIR-NEIST and Assam Science Society Jorhat branch. The Poster was presented on 26th June, 2018 at CSIR-NEIST. Title of the poster was **“Characterisation and Physico-chemical Treatment of Coir Pith to Improve its Hydrophobic and Oleophilic Character for Sorption of Oil Spillage”**.

Authored by Bishnu Sahu, Dipankar Neog, Lakshi Saikia, Dhanjit Das and Jayanta Jyoti Bora

2. Another Poster was presented on 21st February, 2020 in the **International Conference on Engineering Sciences & Technologies for Environmental Care (ESTEC-2020), February 20-22, 2020** organised by CSIR-NEIST, JORHAT. Title of the Project was **“Flow Behaviours Through the Physico-Chemically Modified Coir Dust Based Mat for Application of Prevention of Oil Spillage”**.

Authored by Bishnu Sahu, Dipankar Neog, Dhanjit Das and Jayanta Jyoti Bora.

The poster presentations were shown below:



Characterisation and Physico-chemical Treatment of Coir Pith to Improve Its Hydrophobic and Oleophilic Character for Sorption of Oil Spillage



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Introduction

Petroleum is the soul of any modern industrial society. It is the paramount source of energies and raw material for synthetic polymers and chemicals. But during the processing of crude oil exploration to petroleum products, transportation and refining it creates different types of environmental pollution.

For oil spill cleanup, sorbent is used for preventing the pollution or rectifying the tainted land or water. Sorption is the famous technique for cleanup of oil spills. Coir pith, a natural organic sorbent and a waste product generating from the coconut husk and fibres extraction, could be used for cleanup oil spill which is environment-friendly, biodegradable and economical material with compared to the synthetic materials which are used as sorbent.

Objectives & Methods

Study on the characterisation of coir pith such as calorific value, moisture content, ash content, cellulose content, lignin content, pH value, alkali solubility (1 % NaOH solution), cold water solubility, hot water solubility, bulk density, porosity, water holding capacity etc according to standard methods (TAPPI, 1980; AOAC, 1975) shown in Table 1.

Study different Physico-chemical treatments of coir pith as an absorbent to be used for prevention of oil spillage effect with reference to NE oil industries.

For improving hydrophobicity and oleophilic character of coir pith, physico-chemical treatments such as heating at relative low temperature, acid treatment, alkali treatment, acetylation and acylation treatment were done and absorption in Water and oil were tested as per ASTM method. The properties of treated coir pith were characterized by FTIR and SEM.

Results & Discussions

Table 1. Characterization of Coir pith

Assay	% Content in coir pith/ Value	
	Source 1	Source 2
Calorific value	3820 kcal/kg	3825 kcal/kg
Ash content	13.27 %	13.35 %
pH of coir pith in water	6.64	6.65
Hot water solubility	13.9 %	14.3 %
Cold water solubility	10.5 %	11.2 %
Solubility in dil. Alkali (1%)	17 %	17.2 %
% Swelling of coir pith in H ₂ O	65.88 %	65.72 %
Cellulose content	36.36 %	35.9 %
Lignin content	44.9 %	44 %
Bulk density	156.39 kg/m ³	155.20 kg/m ³
Total porosity	74.4 %	75.75 %
Aeration porosity	18.8 %	20.05 %

Table 2. Particle Size Distribution of 1kg of Coir pith

Average particle Size (mm)	Weight of coir dust (gm)	Weight %
+10	30	3.0
-10 to +5.6	72	7.2
-5.6 to +4.75	70	7.0
-4.75 to 2.36	110	11.0
-2.36 to +1.18	196	19.6
-1.18 to +0.600	236	23.6
-0.600 to +0.300	222	22.2
-0.300	64	6.4

Table 3. Absorption of water and crude oil by different treated & raw coir pith

Size of coir dust +0.600mm	Raw Coir pith	Heat treated Coir pith	Acid treated Coir pith	Acetylated Coir pith	Alkali treated Coir pith
Weight absorbed by Coir pith by water absorption (gm/gm of sorbent)	6.9824	6.1521	7.0780	5.4339	5.0994
Weight Absorbed by coir pith by crude oil absorption (gm/gm of sorbent)	6.0310	6.8351	7.6750	7.7777	9.0617

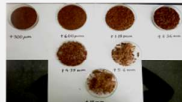


Fig. 1 Particle Size Distribution of Coir pith

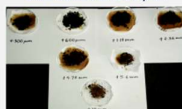
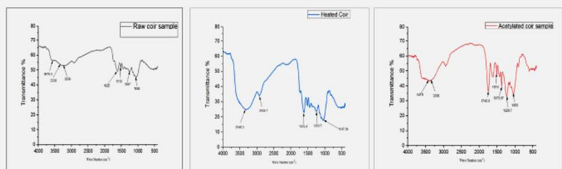
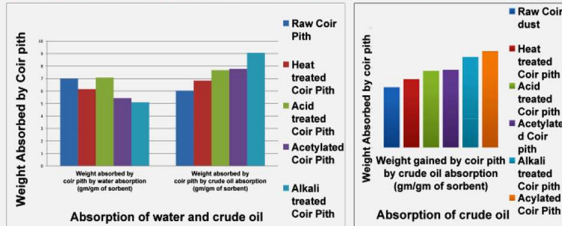


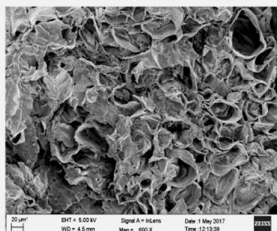
Fig. 2 Crude oil Absorption by coir pith particle

Absorption of Water and Crude oil by raw & treated coir pith particles

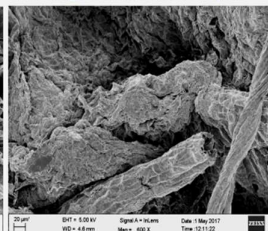


The FT-IR results indicate that coir pith has undergone modification by means of acetylation reaction. The decrease in the intensity of the O-H group absorption bands at 3419cm⁻¹ in spectrum of acetylated coir pith indicated that the acetylation was occurred to the O-H groups. This means hydrophobic character increases in acetylated coir pith.

SEM Image of Acetylated coir pith



SEM Image of heat treated coir pith



- The coir pith sorbent was characterized by standard methods (TAPPI,1980; AOAC,1975) and the results of two sources of coir pith (Source 1 from Biswanath Chari and source 2 from Nalbari, Assam) are shown in Table 1.
- Particle size distribution of coir pith shows that -1.18 mm to +0.300 mm particles are maximum present in coir pith.
- The absorption graph shows that water absorption capacities were lower from 6.98 g-water/g-sorbent to 6.15, 5.43 and 5.09 g-water /g-sorbent for heated coir, acetylated coir and alkali treated coir pith respectively, while crude oil absorption is maximum in acetylated coir pith from 6.03 g-oil/g-sorbent to 9.63 g-oil/g-sorbent, in alkali treated coir pith it is 9.06 g-oil/g-sorbent and in acetylated coir pith it is 7.78 g-oil/g-sorbent.
- Sem image of acetylated coir pith indicates the empty pores which means the absorption capacity increases.

Conclusions

- The hydrophobicity of the Coir pith increases by heating, acetylation, alkalization and acylation, while the absorption of crude oil is maximum in Acetylated coir pith.
- Coir pith was Successfully esterified using oleoyl chloride by the replacement of -OH groups of polymer backbone with that of acyl groups so as to achieve hydrophobic behavior to interact highly with the similarly hydrophobic oil molecules.
- FTIR and SEM characterization were used to investigate the surface morphology and chemical compositions acetylated coir pith. Based on this result, it is undoubtedly true that the modified coir pith could be used for the cleanup of oil spilled in aquatic environments.
- Further studies are need for the design and development of coir pith captr in the shape of membrane/blanket/sheet/pad/block to deal with oil spillage.



Acknowledgement

Authors are thankful to the Director of CSIR-NEIST, Jorhat Assam and also thankful to Coir Board (Min. of MSME) Govt. of India (Project no. CLP-0285) for financial support.

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Figure 27: MRSI poster presentation



FLOW BEHAVIOR THROUGH THE PHYSICO-CHEMICALLY MODIFIED COIR DUST BASED MAT FOR APPLICATION OF PREVENTION OF OIL SPILLAGE

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INTRODUCTION

Petroleum is the soul of any modern industrial society. It is the paramount source of energies and raw material for synthetic polymers and chemicals. But during the processing of crude oil exploration to petroleum products, transportation and refining it creates different types of environmental pollution.

For oil spill cleanup, sorbent is used for preventing the pollution or rectifying the tainted land or water. Sorption is the famous technique for cleanup of oil spills. Coir pith, a natural organic sorbent and a waste product generating from the coconut husk and fibres extraction, could be used for cleanup of oil spill which is environment friendly, biodegradable and economical material with compared to the synthetic materials which are used as sorbent.

Particles Size distribution of the coir pith, size wise absorption, characterization of coir pith was recorded.

Coir dust/pith was Physico-Chemically Modified to particles, blocks and mats for cleanup of oil spillage and checked the absorption in water, crude oil and other oils.

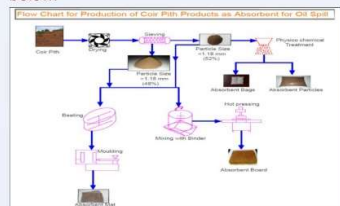
METHODS

Study on the characterisation of coir pith such as calorific value, moisture content, ash content, cellulose content, lignin content, pH value, alkali solubility (1 % NaOH solution), cold water solubility, hot water solubility, bulk density, porosity, water holding capacity, etc according to standard methods (TAPPI, 1980; AOAC, 1975) shown in Table 1.

Permeability of water flow in coir pith particles and mat are studied using Darcy's law.

Study different Physico-chemical treatments of coir pith as an absorbent to be used for prevention of oil spillage effect with reference to NE oil industries.

Flow Chart for Production of coir pith products as Absorbent for oil spill shown below:



RESULTS & DISCUSSION

Table 1. Characterization of Coir pith

Assay	% Content in coir pith/ Value	
	Source 1	Source 2
Calorific value	3820 kcal/kg	3825 kcal/kg
Ash content	13.27 %	13.35 %
pH of coir pith in water	6.64	6.65
Hot water solubility	13.9 %	14.3 %
Cold water solubility	10.5 %	11.2 %
Solubility in dil. Alkali (1%)	17 %	17.2 %
% Swelling of coir pith in H ₂ O	65.88 %	65.72 %
Cellulose content	36.36 %	35.9 %
Lignin content	44.9 %	44 %
Bulk density	156.39 kg/m ³	155.20 kg/m ³
Total porosity	74.4 %	75.75 %
Aeration porosity	18.8 %	20.05 %
Water holding capacity	55.6 %	55.7 %
Permeability of coir pith particles	0.0424 cm/s	0.0343 cm/s
Permeability of coir pith absorbent Mat	0.0161 cm/s	----

Table 2. Particle Size Distribution of 1kg of Coir pith

Average particle Size (mm)	Weight of coir dust (gm)	Weight %
+10	30	3.0
-10 to +5.6	72	7.2
-5.6 to +4.75	70	7.0
-4.75 to 2.36	110	11.0
-2.36 to +1.18	196	19.6
-1.18 to +0.600	236	23.6
-0.600 to +0.300	222	22.2
-0.300	64	6.4



Fig: 1 Particle Size Distribution of Coir pith

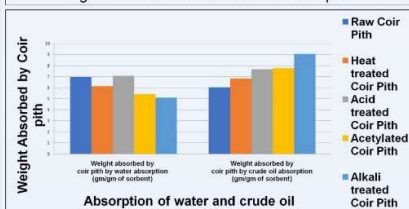
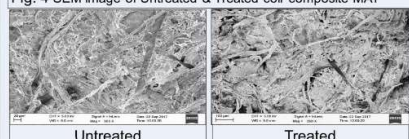


Fig: 2 Absorption of Water and Crude oil by raw & treated coir pith particles

Fig: 3 Absorption of crude oil by Coir dust composite MAT



Fig: 4 SEM image of Untreated & Treated coir composite MAT



CONCLUSION

The permeability behaviors of the coir dust particles and coir dust composite mat were studied and they were sufficient for vertical drains. The permeability reduces in case of coir composite mat.

Characterization of coir pith shows the physical and proximate analysis of coir pith

Particle size distribution of coir pith shows that -1.18 mm to +0.300 mm particles are maximum present in coir pith.

For improving hydrophobicity and oleophilic character of coir pith, Physico-chemical treatments were done and absorption in Water and oil were tested as per ASTM method.

From the SEM Analysis of untreated and treated coir-composite Mat, it is seen that the wax-gum particles were removed in treated of coir dust and the pores of the coir dust were empty. So the absorption capacity of treated coir dust mat increases as compared to untreated coir dust mat.

Product developed for oil absorptions : Coir pith absorbent particles, boards and mats



As coir wastes make some environmental problems, here it is recommended to use them as oil absorbent for prevention of oil spillage, filling material to vertical drains and waste water problems due to its easy modification, abundance and biodegradability

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- Wan Fazilah Fazil Ilahi* & Desa Ahmad A Study on the Physical and Hydraulic Characteristics of Cocopeat Perlite Mixture as a Growing Media in Containerized Plant Production. Sains Malaysiana 46(6)(2017): 975-980
- U.P. Nawagamwa1* and G.H.A.J.J. Kumara2* Utilization Of Coir Dust To Partially Replace Sand In Vertical Drains For Soft Ground Improvement. International Conference on Sustainable Built Environment (ICSBE-2010) Kandy, 13-14 December 2010
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- V Barlianti and El Wiloso, Akta Kimindo, 2007, 3, 21-26.
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Figure 28: ESTEC 2020 poster presentation

Paper Publication:

“Development of a high oil absorbent mat from coir dust and evaluation of their physico-chemical properties”, Pallav Saikia, Dipanka Dutta, Dipul Kalita & Jayanta Jyoti Bora (Under review)

Technology Developed: “High Oil Absorbing Mat/Pad from Coir Waste”

1. **Raw Materials :** Coir waste, Natural fibre, Natural binder
2. **Production Capacity :** 60,000 sheets per annum
3. **Application/Uses:**
 - The product is used in oil spilling related to oil exploration, industries, transportation etc.
 - The product can be used in machine/vehicle maintenance work.
4. **Brief technical details:** Absorbing Mat/Pad is made from the mixture of coir waste and natural fibre using a natural binder. The Mat/Pads are used in oil spilling related to oil exploration, industries, transportation etc. Also product can be used in machine/vehicle maintenance work. The product has higher oil absorbing property, reliable tensile strength with flexibility, good tear & smooth surface, better to commercial products.
5. **State of Art:** Coir pith/dust, is one of the solid wastes generated from coir industry and are usually dumped nearby open grounds causing soil and air pollution. These can be converted to value added product by adopted certain mechano-chemical processing. There are some other wastes generated in our day to day activities such as gunny bag/jute waste and waste cotton etc, which can also be effectively utilized with coir wastes for to making useful product. The process for making oil

absorbing Mat/Pad is economic and free from the use of hazardous chemicals. Thus the product is eco- friendly. The use of natural fibre with coir pith/dust in order makes the composite sheet more eco- friendly and durable found suitable for using in oil spilling related to oil exploration, industries, transportation and used in machine/vehicle maintenance work.

6. Innovative Components:

6.1 Patent applied /filed/granted on the technology (if any): 1 Patent applied

6.2 Publication communicated/published on the proposed technology (if any):

Publication communicated: 1 No.

Poster Presentation : 1 No.

6.3 Briefly indicate the innovative component of the process/technology proposed : Oil High oil absorbing Mat/Pad from coir wastes is a new product which is durable and eco- friendly. No effluent/gas generates during the manufacturing of the product. Natural fibre is used to make the product more absorbent, eco-friendly and more durable. A natural binder material used in the process gives higher flexibility, smoothness and more durability.

7. Effluents and the Environmental Consideration: No effluents generates during the manufacturing process. Hence the process is environment friendly.

8. Health consideration and toxicity (if any): The ingredients used in the process are non-toxic. Hence the product doesn't have any negative effect on human being

9. Requirement of approval needed from statutory bodies (if any): --

10. Level/scale of Development : Industrial scale level

11. Industrial Requirements:

11.1 Comments on reproducibility: One time use.

11.2 Thermal and Chemical risk and hazards (if any): Nil

11.3 Safety issues: 100% safe.

Bio-safety considerations, if applicable: Eco-friendly product.

12. Basic raw materials: Coir waste- coir pith/dust, natural fibre and natural binder

13. Major plant and machineries required:

Major plant and machineries required are as follows:

- i. Shedder
- ii. Valley Beater
- iii. Handmade Vat
- iv. Press machine
- v. Weighing balance
- vi. Miscellaneous

14. Cost benefit analysis:

14.1 Unit capacity of the process/technology proposed for transfer: 2 sheets per day or 60,000 sheets per annum

14.2 Tentative project cost for the proposed capacity: Rs. 18.8 Lakhs
(Please see project profile in TABLE-I)

14.3 Profitability: Approx. gross profit 11.95 Lakhs per annum (Please see project profile in TABLE-VI)

14.4 Break-even analysis: 25.64% (Please see project profile in TABLE-VI)

Brief economic impact assessment justifies the proposed technology vis-a-vis present market demand:

High Oil absorbing Mat/Pad is a new product made from coir pith/dust a waste generated from coir industry. Such waste materials are usually damped nearby industrial site which ultimately creates soil or air pollution to a greater extent. The use of such waste material not only reduces environmental pollution but also helps in making high oil absorbing mat/pad for oil spill. Oil spill is one of the major sources that contribute to environmental pollution in oil Industry Due to the gradual increasing environmental pollution in oil industry, hence there is need of control this pollution. The high oil absorbing mat/pad developed by CSIR-NEIST has some speciality for which is suitable for using in oil spilling related to oil exploration, industries, transportation. It can be used in machine/vehicle maintenance work, has also higher market potentiality. Hence, establishment of such industries may create employment opportunity and helps income generation.

15. Brief SWOT analysis of the technology:

Strengths	Weaknesses	Opportunities	Threats
<ul style="list-style-type: none"> • Technology of similar type is presently available at CSIR-NEIST • Low cost, eco-friendly technology • Having expertise in composite making • Availability of machinery and equipment at CSIR-NEIST 	<ul style="list-style-type: none"> • Non availability of sufficient coir waste (Raw material) in the NE region 	<ul style="list-style-type: none"> • The product has high market potentiality • Possibility to set up industry small/medium scale • Availability of natural fibre material 	<ul style="list-style-type: none"> • Raw materials/ Chemicals required for the technology are to be procured from the market • Synthetic absorbent mats are available in the market

PROJECT PROFILE ON HIGH OIL ABSORBING MAT/PAD FROM COIR WASTE

1. <i>Product</i>	:	High Oil Absorbing Mat/Pad From Coir Waste
2. <i>Raw Materials</i>	:	Coir waste, Natural fibre, Natural binder
3. <i>Production Capacity quality</i>	:	60,000 Sheets per annum
4. <i>Project Cost</i>	:	Rs.18.800 Lakhs
5. <i>Break-Even Point</i>	:	25.64 %
6. <i>Know How Fee</i>	:	Rs. 2 Lakhs
7. <i>PI</i>	:	Mr. Jayanta Jyoti Bora ¹
<i>Co- PI</i>	:	Dr. Dipul Kalita ²
<i>Team Members</i>	:	Mr. Pallav Saikia ² Mr. Dipankar Neog ¹ Dr. Tridip Goswami ² Mr. Subodh Chandra Kalita ¹
¹ General Engineering Group, Engineering Sciences & Technology Division		
² Cellulose Pulp & Paper Group, Material Sciences & Technology Division		
CSIR- North East Institute of Science & Technology, Jorhat, Assam, Pin: 785006		

INTRODUCTION

Oil spill is one of the major sources that contribute to environmental pollution in oil Industry. It happens as a consequence of activities related to crude oil exploration, industries, and transportation. On the land, oil spill penetrates to soil matrix and thereby contaminate ground water. The entire process of oil spill pollution is handled worldwide by two ways, restricting oil spill from spreading to the surroundings and removing the spill from contaminated surroundings. Coir pith/dust, a waste generated from coir industry and a natural organic sorbent, is an environmental-friendly, biodegradable, renewable, cheap material and has got excellent chemical and physical properties, which makes it suitable for diverse applications. Since a major portion of this waste is biological origin, hence the use of these materials in making value added products would definitely be helpful from the point of ecology and environment. These solid waste materials alone or in combination with natural fibres may produce useful products through certain mechano- chemical processing. Plant fibres are being used for several generations in man made products/processes due to their easy availability, inexpensive, low density, low energy consumption, biodegradability and renewability nature. There is also a trend that the products made from biological origin are considered to be environmentally benign. They exhibit several advantageous properties like better mechanical strength, lower density, oil absorbing property, yielded light weight flexible composite materials which find use in many industrial applications. Thus incorporating bio-renewable materials makes the final product biodegradable, renewable and more eco-friendly.

A numbers of steps are involved in the preparation of oil absorbent mat using coir pith/dust as raw material. Some important steps include disintegration of the dust materials, mixing with suitable binder, sheets making in handmade vat to get required shape & size, drying of the sheets and final product making.

Considering the gradual increasing environmental pollution by oil spilling in oil Industry, CSIR-NEIST has developed a highly oil absorbing mat/pad from coir industry wastes i.e. coir pith/dust in combination with natural fibre i.e. jute/ cotton fibre using natural binder. Utilization of such waste materials is not only help to reduce environmental pollution, but also help for making a product suitable for using in oil spilling related to oil exploration, industries, transportation and in machine/vehicle maintenance work.

SALIENT FEATURES:

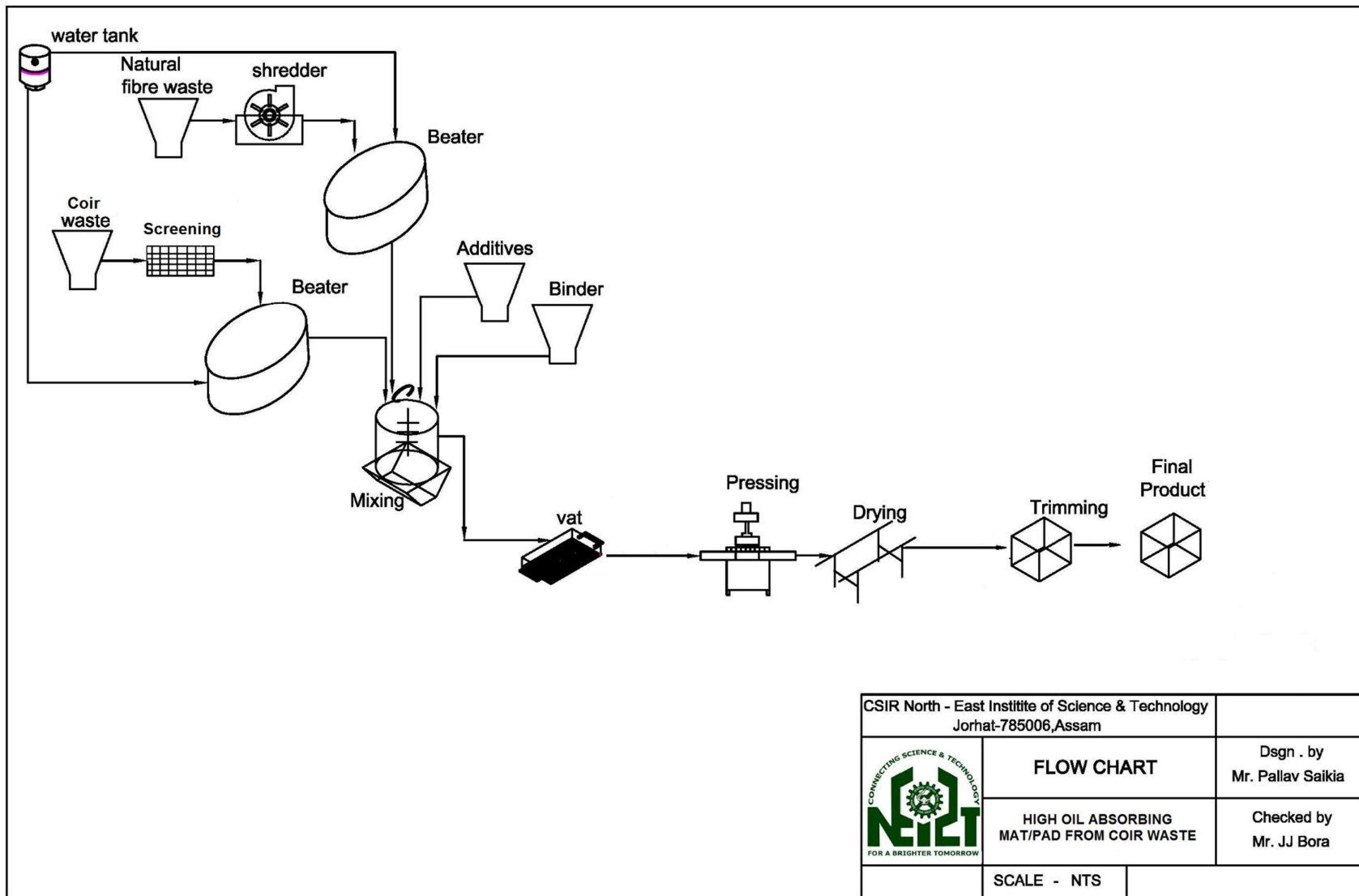
- Use of coir waste and natural fibre for making useful product.
- Recycling of waste material reduces environmental pollution.
- Simple, eco friendly technology with zero discharge.
- Use of solid waste reduces the environment pollution.
- The technology is simple and the final product is eco-friendly.
- Suitable for small/cottage scale industry also.

ADVANTAGES OF THE PRODUCT:

- The product is highly oil absorbing capacity and possesses higher mechanical strength properties.
- Use for oil spilling related to oil exploration, industries, transportation.
- The product is found suitable to use in machine/vehicle maintenance work.
- The product is chipper water repellent, flexible and smooth.

PROCESSING STEPS:

- Cleaning and defibration of the coir waste and natural fibre.
- Mixing and treatment with special chemical of the coir waste and natural fibre.
- Sheet making and pressing of the sheets.
- Sun drying or oven drying of the sheets.
- Trimming and packaging of finished product.



USES:

- The product is used in oil spilling related to oil exploration, industries, transportation etc.
- The product can be used in machine/vehicle maintenance work.

TECHNO- ECONOMIC:

The investment cost for production of 200 sheets per day capacity plan will be approximately Rs. 18.80 Lakhs.

TABLE- I
PROJECT CAPITAL COST

Sl. No.	PARTICULARS	Amount (Rs.)
A	MACHINERY	12,95,000.00
	Shredder, Valley Beater, Handmade Vat, Screw press/Hydraulic press, Weight Balance, Miscellaneous, Electrical Fitting & Lighting, Office furniture	
B	PRE-OPERATIVE EXPENSES	50,000.00
C	MARGIN MONEY FOR WORKING CAPITAL	3,10,000.00
D	KNOW HOW FEE + CONSULTANCY FEE	2,00,000.00
E	GST 12% of D	24,000.00
GRAND TOTAL		18,79,000.00

TABLE - II

COST OF PRODUCTION

Number of Working Days =300

Plant Capacity = 200 Sheets per Day

60000 Sheets per annum

Sl. No	Item	Requirement		Price (Rs)		Amount (Rs)
1	Raw Material Cost					
	Coir Pith	92.79	Kg per day	8.00	per Kg	2,22,693.82
	Natural fibre	16.37	Kg per day	30.00	per Kg	1,47,370.91
	Binder	10.92	Kg per day	100.00	per Kg	3,27,490.91
	Beater Additive	1.20	Kg per day	6.00	per Kg	1,440.96
	Raw Material Cost					6,98,996.60
2	Manpower Cost	As per Table III				6,18,000.00
3	Utility					
	Electricity	35	units per day	5.71	per unit	59,955.00
	Water	1000	litre per day	2.50	per 100 litre	7,500.00
	Utility Cost					67,455.00
4	Packaging Cost	200	sheets per day	0.25	per sheet	15,000.00
5	Maintenance & Repair	5%	of Machinery			64,750.00
6	Depreciation	10%	of Machinery			1,29,500.00
	Depreciation Cost					1,29,500.00
7	Interest on Bank Loan	10.5 %	of Bank Loan			1,97,295.00
8	Miscellaneous Expenditure	2%	of Raw material Cost			13,979.93
TOTAL COST OF PRODUCTION						18,04,976.53

SALES REALIZATION

Sl. No	Item	Quantity		Selling Price (Rs)	Amount per Annum (Rs)
1	Oil Absorbent Mat/Pat	200	Sheets per Day	50.00 per sheet	30,00,000.00
TOTAL SALES REALIZATION					30,00,000.00

TABLE - III

Cost of Manpower

Sl. No.	Type	Quantity	Wage/ Monthly Salary in Rs.	No. of Days or month		Amount in Rupees
1	Production Manager	1	9000.00	12	months	108000.00
4	Labour	6	250.00	300	days	450000.00
5	Watchman	1	5000.00	12	months	60000.00
Total Yearly Cost						618000.00

Table - IV

Margin Money for working capital

Sl. No.	Particulars	No. of Months		Amount in Rupees
1	Raw Materials	2	Months	116499.43
2	Manpower Cost	2	Months	103000.00
4	Utility	2	Months	11242.50
5	Packaging Cost	90	days	4500.00
5	Maintenance & Repair	2	Months	10791.67
6	Depreciation Cost	2	Months	21583.33
7	Interest on Bank Loan	2	Months	32882.50
8	Miscellaneous Expenditure	2	Months	2329.99
TOTAL				302829.00
Say				310000.00

PROFITABILITY ANALYSIS

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TABLE - VI

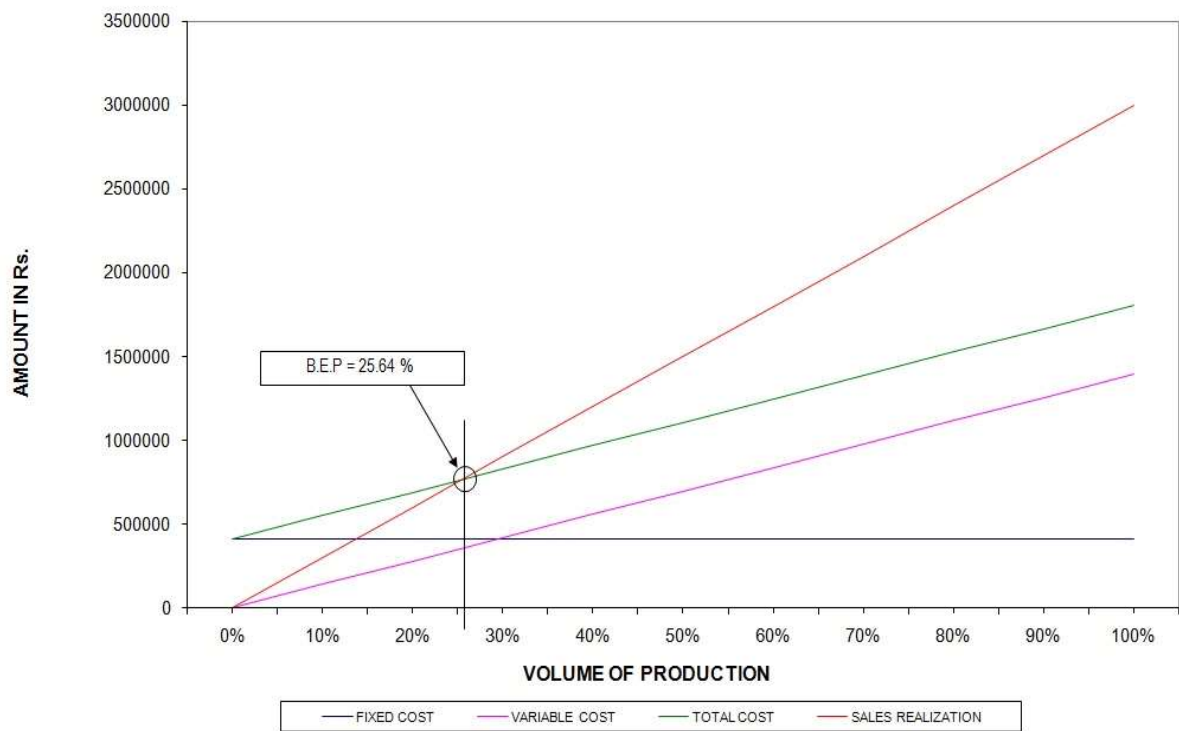
BREAK-EVEN ANALYSIS

Sl. No.	Particulars	Amount in Rupees
A	FIXED COST	
1	Manpower Cost	60000.00
2	Interest on Bank Loan	197295.00
3	Utility	9542.39
4	Depreciation	129500.00
5	Maintenance & Repair	12950.00
6	Miscellaneous Expenditure	2795.99
	TOTAL OF (A)	412083.37
B	VARIABLE COST	
1	Raw Material Cost	698996.60
2	Manpower Cost	558000.00
3	Utility	57912.61
4	Packing Cost	15000.00
5	Maintenance & Repair	51800.00
6	Miscellaneous Expenditure	11183.95
	TOTAL OF (B)	1392893.15
C	TOTAL COST (A + B)	1804976.53
D	SALES REALIZATION	3000000.00
E	GROSS PROFIT	1195023.47
F	BREAK - EVEN POINT	25.64%

COST OF PRODUCTION PER SHEET 30.10

SELLING PRICE PER SHEET 50.00

BREAK-EVEN ANALYSIS





High Oil Absorbing Mat/Pad



Honourable Union Minister of Road Transport & Highways and MSME, Govt. Of India Sri Nitin Jairam Gadkari launched technology on **Oil Absorbing Mat from Coir Pith** which is developed under Collaborative Project “**Development of Appropriate Product by Studying the Possible Use of Coir Dust in Oil Industries with Reference to North-East India for Absorption of Oil Spill**” of CSIR-NEIST & COIR BOARD on 28th September, 2019 in the Inaugural function on the new initiatives of COIR BOARD at Vellore, Tamilnadu.