Physicochemical Characterization of Cattle Dung Fibre Under the Hydrothermal Process

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Research Article

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Abstract

Cattle dung fibre is a lignocellulosic material abundant and sustainable non-wood source of polymeric components, which can be converted into high added-value products. Hydrothermal treatment of the fibre obtained from cow dung was explored using four different temperatures (120, 140, 160 and 180 °C) and incubation times (0 and 120 minutes) at a fixed material to water ratio (1:10). The present study resulted in the highest yield of 94% (w/w) that gradually decreased with temperature and incubation time. The physicochemical analysis revealed that hydrothermal treatment resulted in high cellulose, low lignin, and ash content (51.6%, 30.93%, and 6.3%, respectively) at 160◦C for 2 hr incubation time and was appropriate for pulp and paper production. The SEM and X-ray crystallography indicates the treatment resulted in separated fibrils and a porous structure. Both FTIR studies and chemical characterisation techniques were used to optimize the temperature and duration of hydrothermal treatment. Overall, the study presents the first report on the extraction of fibres from cow dung and their hydrothermal treatment. In perspective, it is possible to achieve the properties required for its industrial-scale conversion to eco-friendly papers by heating the fibre under controlled conditions.

Introduction

Paper plays a significant role in our daily life. Due to the increasing human population, the demand for paper and its products is increasing daily. The global annual paper and cardboard production was reported to be more than 400 million metric tons in 2020, with China and United States as the primary paper producing countries. However, the said demand for paper is higher than the overall global paper production capacity (Statista report 2020a). Various types of wood (90%) and plant materials (10%) are used to prepare paper, ultimately diminishes forest resources. To reduce the environmental impact, deforestation and maintenance of circular economy, it is imperative to look for alternative materials for the generation of fibre and pulp. Many of the commonly available sources of fibre are generally undervalued and ignored. Agricultural waste, and livestock-based waste or bio-residue, are potential sources of fibre, especially in any agricultural-driven country.

The global livestock population is around 987.51 million heads, with India’s share as 303 million heads, followed by Brazil, the United States, and China in 2020 (Statista report 2020b). India also had the most significant number of cows in the world in 2019. The rearing of many cows results in producing a large amount of cow dung. Various bovine-dairy based industries find it challenging to manage such a large amount of generated animal dung (Kleinman et al., 2019). Researchers have reported that around 2600 million tons of dung is generated and is a source of greenhouse gases like methane and nitrous oxide (Kaur, 2017). This untreated bio-residue, when leached into water bodies and can contaminate the groundwater as well. There is a strong need to develop techniques to valorize this waste material and convert it into value-added products that can help drive the economy of rural and urban areas.

Although manure production from cow dung is already a well-established technique worldwide, it remains unexplored in the domain of fibre extraction, which has tremendous potential. Cattles generally chew food through the cuddling process, and undigested food is ejected out through excreta. It has been found that around 70–80% of cellulose becomes a part of dung. Studies have shown that fibre with an average length

of 0.8-1.3mm and a 900 to 985 (μm) diameter can be extracted from cow dung (Fasake and Dashora, 2021a). The presence of a naturally aligned carbon-carbon bond can endow these naturally occurring fibres isolated from bio-residue with high strength and stiffness (Jústiz-smith et al., 2008). The overall qualities of this fibre is a vital function of the type of animal species, age, and eating habits (Zhu et al., 2020).

Researchers have initiated studies on the extraction of fibres from two different types of Indian-breed cows, i.e. Indigenous cow, a Jersey cow, and a buffalo. The amount of cellulose (29-31.50%) obtained from these bio-residues is comparable to wheat straw (~ 39%), indicates its potential to be used in the production of paper (Fasake and Dashora, 2020b). High cellulose in the biomass furnishes it with suitable properties to make the form strong. In the paper industry, lignin is an undesirable component in the pulp and needs to be removed using a suitable reagent (Kaur et al., 2019). Removal of lignin using chemical and mechanical means can help utilize this pulp to prepare paper and various innovative products like pots, cartons and trays, etc. (M'Hamdi et al., 2017). Hydrolysis, a hot-water treatment (150-250^{0}C), is used to solubilise polysaccharides and increase the cellulosic fraction. The generation of acetic acid by hydrolysis of the acetyl group is expected to catalyze the hydrolysis of hemicellulose. Lignin is removed, and the residue is enriched with cellulose (Caparrós et al., 2008). This hydrothermal treatment of pulp does not use expensive chemicals and is thus eco-friendly and cost-effective.

Thus, the present study aims to create cow dung fibre and perform its hydrothermal treatment. The chemical characterisation of differently treated samples was performed using glucose, xylose, arabinose, ASL (Acid soluble lignin) and AIL (Acid insoluble lignin), crystallinity and ash content as parameters. Change in surface morphology was investigated using Scanning Electron Microscopy (SEM) analysis. The effect of hydrothermal treatment on the chemical composition of fibre was further investigated using Fourier Transform Infrared Spectroscopy (FTIR) analysis. New green treatment has been proposed for the first time to provide an elaborated outlook for the effective management of cattle dung waste and the successful production of fibre for the paper industry.

Materials And Methods

Sample Collection and preparation

Raw animal dung sample was received from Mandir gaushala, Kishangarh, Maheroli, New Delhi. Under the author's observation, a trained person from gaushala handled animals and the dung collection. The slurry of fresh cow dung was prepared by mixing it with water and subsequently passed through different sieves, as mentioned in (Fasake and Dashora, 2020a). The semi-digested dung fibre particles obtained through BSS 20 and BSS 40 sieve were collected and kept at room temperature for one day with overturned occasionally using sterilized wooden sticks. After attaining the safe moisture level, the dung fibre material was stored in separate airtight plastic bags under dry conditions for subsequent experiments.

Hydrothermal treatments

Hydrothermal treatment is an environmental-friendly technique for treating cattle dung fibre as it does not involve any toxic chemicals. There is no associated equipment corrosion problem with it. Also, the use of

water only makes it an economically feasible process. Figure 1 shows the hydrothermal treatment based cellulose-rich fibre production scheme most suitable for the paper industry.

The hydrothermal treatment was performed in a laboratory-scale batch reactor procured from Amar equipment, Mumbai, India, with a maximum volume of 2000 mL. The 100 g samples were mixed with 1000 mL of deionized water and heated at designated temperatures (120°C, 140°C, 160°C, and 180°C) with different incubation times (0 min and 120 min) with the heating rate of 4°C/min. As the planned condition was achieved, tap water was immediately opened through the stainless-steel cooling ring inside the reactor to reduce the inside temperature to room temperature. Figure 2 shows the picture of the reactor used for the study. The temperature dropped rapidly at the start of the cooling process, and the cooling time varied depending on the pretreatment temperature. After the reaction, the solid fractions were collected by filtration and thoroughly washed to the neutral pH. The yield percentage was calculated by the initial weight of the sample divided by the final weight.

Chemical Characterization

The chemical composition of the raw dung fibres and hydrothermally treated (HT) samples were determined and compared. The content of sugars in the solid dung fibres was measured after substantial acid hydrolysis of the polysaccharides. 300 mg milled samples were treated with 3 mL 72 % (v/v) H_2SO_4 at 30°C for 1 hour. The hydrolysate was diluted with 84 ml distilled water and autoclaved at 121°C for 1 hour. The solution was filtered through crucibles. The aliquot will be used to determine acid-soluble lignin (ASL) and carbohydrate. The Acid insoluble lignin (AIL) was determined based on the filter cake subtracting the ash content after incineration at 575°C for 6 hrs using the National Renewable Energy Laboratory (NREL Protocol, CO, USA, 2012). The content of sugars in the liquid extract was measured after acidic hydrolysis of the polysaccharides. The High-performance liquid chromatography (HPLC) analysis was performed (Agilent 1200 series, Agilent Technologies, Santa Clara, USA) with an Aminex HPX-87P column at 60°C. The 5mM sulfuric acid (5mM) was used eluent at the flow rate of 0.5 ml/min. The components were detected using a RI detector. Ash content was determined by TAPPI -T 211 cm-02 standards method.

Scanning Electron Microscopy (SEM)

The microstructural changes like surface texture and porosity of the sample were characterized by scanning electron microscopy (SEM). The raw and treated material was first converted into a fine powder and then observed on a model TM-3000 scanning electron microscope (HITACHI, Japan). The fibre samples were placed randomly over the silver tape, sprayed and fixed pieces on a thin gold coating (Emitech K550X) to minimize its charging because of non-conducting materials. Images were taken at several magnifications ranging from 100× to 6000×.

Fourier Transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared Spectroscopy studies were performed to understand the change in functional groups in the sample before and after fibre hydrolysis. The analysis of the powdered dried sample was performed using Nicolet iS10 FTIR system (Thermofisher Scientific, Altham, MA, USA). All the spectra were recorded in 400–4000 cm^{-1} wave number.

XRD analysis

The crystalline structure of cellulose was determined by X-ray diffraction analysis (Rigaku Ultima IV, Ri, Tokyo, Japan). Both samples were scanned at the diffraction angle ranging from 3 to 60^0 with a scan speed of 2^0/min. The crystallinity index (CrI) was calculated according to (Segal and Martin, 1959).

$$CrI\% = \frac{I_{002} - I_{AM}}{I_{002}} \times 100$$

I_{002} and I_{AM} are the intensity of crystalline and amorphous phase localized at 22° and 18° in 2θ.

Statistical Analyses

The experiments were repeated in triplicate, and the results are reported as the mean of replicates with standard deviation (mean ± SD) of the values.

Results And Discussion

Chemical Characterisation

The primary constituents of biomass include cellulose (~ 50%), hemicellulose (~ 25%), lignin (~ 25) (Kaur et al., 2016). The proportion of these components varies with the type of plant species and the geographical location of their growth (Casey 1980). A high proportion of lignin is undesirable for pulp and paper. The initial chemical composition in control was found to be: 37% cellulose (estimated as glucose), 22.8% hemicellulose (including 20.1% xylose and 2.7% arabinose) and 6.2 ± 0.12% ash content. The values are comparable to the reported cellulose, hemicellulosic of wheat straw as 35.10% and 25.60%, respectively (Chandra et al., 2012).

Hydrothermal treatment of raw material was performed by placing it in a reactor surrounded by an aqueous medium at an elevated temperature and pressure. According to Liu et al., 2012 the temperature below 100 °C has no hydrolytic effect on the material, and at above 220 °C, cellulose degradation occurs. Thus, the present study was planned in a temperature ranges between 120 to 180 °C for 0 min and 120 min reaction time. After conducting a preliminary optimization experiment, the material to water ratio (1:10) has finalized according to the apparent and bulk density (Fasake and Dashora, 2021c).

Table 1
chemical characterisation of hydrolysed sample and raw material

Reaction Temp. (°C)	Reaction time (Min)	Yield[a] (%)	Glucose (%)	Xylose (%)	Arabinose (%)	ASL (%)	AIL (%)	Crystallinity (CrI%)	Ash (%)
Untreated (Raw sample)		100	37	20.1	2.7	1.3	24.7	41	6.2 ± 0.12
120	0	93 ± 1.38	38.9	20.7	3.4	0.91	25.63	39	6.0 ± 0.21
	120	90 ± 1.15	38.9	21.0	3.3	0.85	28.30	37	6.1 ± 0.16
140	0	88 ± 1.44	42.3	22.2	3.5	0.95	27.18	47	5.8 ± 0.11
	120	82 ± 1.32	42.0	22.5	2.9	0.81	27.21	38	7.2 ± 0.25
160	0	87 ± 0.98	42.8	23.2	3.0	0.86	28.92	35	7.0 ± 0.28
	120	68 ± 1.11	51.6	17.8	0.9	0.94	30.93	46	6.3 ± 0.14
180	0	67 ± 1.61	60.3	5.2	0.0	1.01	32.71	49	6.4 ± 0.22
	120	62 ± 1.36	60.2	5.2	0.0	1.22	38.08	48	7.6 ± 0.17

[a]Weight % based on the starting materials, AIL: Acid insoluble lignin; ASL: Acid soluble lignin.

Table 1 shows the effect of hydrothermal treatment on the chemical characteristics of raw material. The maximum yield of 93% was obtained at the temperature of 120^0C with no holding time. It was found to decrease with a further increase in temperature or contact time. The increase in contact time did not help in improving yield. The raw sample exhibited glucose, xylose, arabinose as 37%, 20.1%, and 2.7%, respectively. No regular pattern on the effect of temperature on xylose and arabinose content was observed.

In contrast, long incubation times noticeably changed the chemical, morphological parameters and yields. The glucose and xylose contents were constantly varied up to 140°C for 2 hr incubation time and then abruptly changed when temperatures higher than 160°C were used. The increase in temperature and contact time resulted in the lowering of xylose and arabinose content. It might be because of the removal of

extractives and hemicellulose fraction by hydrothermal. The treatment of biomass to a higher temperature (180^0C) resulted in low yield (33–38%) and glucose content (60%).

Compared to increasing the reaction temperature, increasing incubation time is more efficient in booming cellulose bioconversion. Similar observations were reported by Yu et al. (2010). As shown in Table 1, the glucose value at 120 °C for 2 hr and 140 °C for 0 hr were almost equal, indicates both the incubation time and temperature had shown a positive effect on hydrothermally treated material under the relatively low temperature (120 °C to 140 °C). However, the increase in cellulose biodegradation can be attributed to the destruction of fibre cell walls due to solubilizing or dissolving lignin, breaking the bond of lignin-carbohydrate connection (LCC), and hydrolyzing the amount of hemicellulosic moieties (Mosier et al. 2005). When the temperature and incubation period was increased, the retained glucose percentage in the material also increased, accompanied by the simultaneous degradation of the hemicellulose carbohydrates (Xylose and arabinose) content. A relative increase in lignin content accompanied the process.

Moreover, the relative increase in cellulose decreased amorphous hemicelluloses, and part of lignin resulted in a gradual rise in crystallinity index (CrI). At 120°C, the crystallinity index of the sample and control is almost the same. At any particular temperature with an increase in treatment time, CrI% decreased. Unusual behaviour at 160°C was observed at 160°C, where CrI% decreased with the temperature rise.

Cellulose is a polysaccharide made up of D-glucose units, joined by β-1-4-glucosidic bonds. There are three free hydroxyl groups present in each anhydroglucose unit (AGU) connected by intermolecular hydrogen bonds, leading to forming two domains of high ordered arrangement (crystalline) and random arrangement (amorphous region). The availability of enzymes, solvents and chemicals to the hydroxyl group and overall reactivity of cellulose is altered. The degree of crystallinity indicates the reactivity level of cellulose present in the compounds (Ferreira et al., 2014). The rise of CrI % value shows that a higher amount of cellulose is available for reaction with increasing temperature and treatment time.

Raw dung material has a porous structure that includes lumens and pits, as shown in Fig. 3. When dung fibres were immersed in the water, initially, the fibre surface was socked. Later, due to penetration, fibre became wet. The capillaries are helpful to fill the void space and the amorphous regions of the cell wall. The sorption of water into the fibre is a complex process. In pulp preparation, water diffuses into the amorphous regions of the cellulose matrix and breaks inter-molecular hydrogen bonds between cellulose surfaces. Swelling fibres increases the volume, while the surface area does not expand (Botkova et al., 2013). This swelling effect increases the inter-molecular distance of the cellulose chains. It also facilitates the diffusion of sugars and oligomers of hemicelluloses to the aqueous medium (Rowell, 2016). The ash content was observed between 6 to 8% in controlled and all hydrothermally treated samples and which is equal and less than the other non-wood materials, such as Bagasse (8.02%), rice straw (20.02%), corn stover (7.82%) etc. Generally, the lower ash content indicates maximum pulp yield with good quality of the paper. Chemicals play a crucial role during the production process of the paper. The soda and kraft process is the most commonly used method for treating wood and non-wood related materials. Making paper with the help of chemical is not an economically feasible process for cattle dung fibre. The lignocellulosic fodder material has processed into cattle's rumen system using bacteria, microbes and get a semi-digested material in the form of fresh

dung. One kg of fresh cattle dung gives 10–12% raw fibre depending on their diet and other factors reported by Fasake and Dashora 2020.

Yield plays a vital role in any processing industry. The chemical method reported resulted in about 50% yield, which means that about half of the dung fibre material was lost during the digestion process at 140°C for 2 hr with only 1% NaOH. In the present studies, the hydrothermal treatment resulted in a yield of 82% at the same temperature and time. Though slightly higher cellulose and lower lignin content were reported in chemically treated samples, composite hydrothermal treated samples are also suitable in the making of paper.

Scanning Electron Microscopy (SEM)

SEM is used to study surface characteristics and change in morphology of fibres after treatment. The SEM images of control and treated fibres are shown in Fig. The external surface exhibited a rough structure, and uneven surface morphology with heavy deposition may be of hemicelluloses, lignin, wax, pectin and inorganic components. It can be seen from Figure 4 that the surface of the control sample was smooth, light coloured and non-porous. In comparison, the treated samples were broken, dark-coloured. With increasing temperature and treatment time, the colour intensity and cracks also increased. The surface of the untreated sample exhibited a porous structure, unlike the non-porous system of control. By visual observations, it was found that as the temperature increased, the treated fibres smelled of burnt sugar, which might be due to Maillard Browning products in C5 sugar degradation and fermentation of peptide amino groups (Liavoga et al., 2007). The lignin component could be melted and amalgamating in the form of tiny granular and spherical droplets on the fibre's surface. However, some flattened disks and irregular droplets could be found due to the reshaping process and nonuniform distribution of lignin in cell walls and the simple aggregation of the hydrophobic lignin in a hydrophilic environment (Donohoe et al., 2008).

FTIR

Spectra of the sample treated at different temperatures and time were recorded, as shown in Figure 5. Two dominant features appear in the region between 500-2000 cm^{-1} and 2200-3700 cm^{-1}. IR spectra between 3000 to 3700 cm^{-1} corresponds to cellulose hydrogen bonds. C-H stretching occurs between 2800–3000 cm^{-1}. C-H bending, C-H scissoring, and C-H2 wagging in cellulose is reported between 1300 and 1400 cm^{-1}. The region below 1300 cm^{-1} shows changes in C-O-C bonding pattern, asymmetric stretching from glycosidic linkages, C-O stretching, C-O-C deformation.

The region between 600 to 1500 cm^{-1} corresponds to cellulose and hemicelluloses. The sharp peaks in this region show the change in the chemical structure of cellulose and hemicellulose on heating. Characteristic bands at 1510, 1595, 1740 and 1770 cm^{-1} are related to the change in lignin content of the pulp on heating to 120^0C for 0 hours. Further heating beyond 120^0C for more and more period brings on a significant shift in lignin structure. The studies suggest heating for 120°C for 0 hours is the most optimum temperature to remove lignin and concentrate the pulp to have higher cellulosic content. Findings are similar to the observations reported in the chemical analysis shown in Table 1. The unusual behaviour of fibres reported for CrI% has observed in FTIR also where peaks crossed each other in the region of 4000-3000 cm^{-1}.

Conclusion

In conclusion, the analysis of the hydrothermal treatment of fibres obtained from cow dung has suggested that lignin removal can be achieved efficiently. No regular pattern of change was observed in the case of xylose and arabinose content with temperature change. The temperature and incubation period increased the retained glucose percentage, which could be due to the simultaneous degradation of the hemicellulose carbohydrates. The decreased amorphous hemicelluloses and lignin contents were also observed. This finding was accompanied by a gradual increase in crystallinity index with temperature. The ash content between 6 to 8% in both controlled and treated samples was less than other non-wood species indicating the maximum pulp yield with excellent paper quality. SEM analysis showed the porous and cracked surfaces of treated samples. Findings of FTIR were similar to the properties observed during chemical characterisation. Heating for 120°C for 0 hours was regarded as the optimum temperature for the maximum lignin removal and enriching the pulp with cellulosic content. Although chemical treatment may result in higher lignin removal efficiency, economic feasibility and environmental sustainability need special attention in that case. Hydrothermal treatment of cow dung fibre is an efficient process as it serves the purpose of valorising waste dung.

Declarations

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Conflicts of interest/Competing interests: The authors declare that they have no conflict of interest**.**

Availability of data and material: Not applicable

Code availability: Not applicable

Ethical statement: No animal or human studies were carried out by the authors. Under authors' observation, collection of dung was done by a trained person from the gaushala.

Ethics approval: Not applicable

Consent to participate: Not applicable

Consent for publication: Not applicable

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Figures

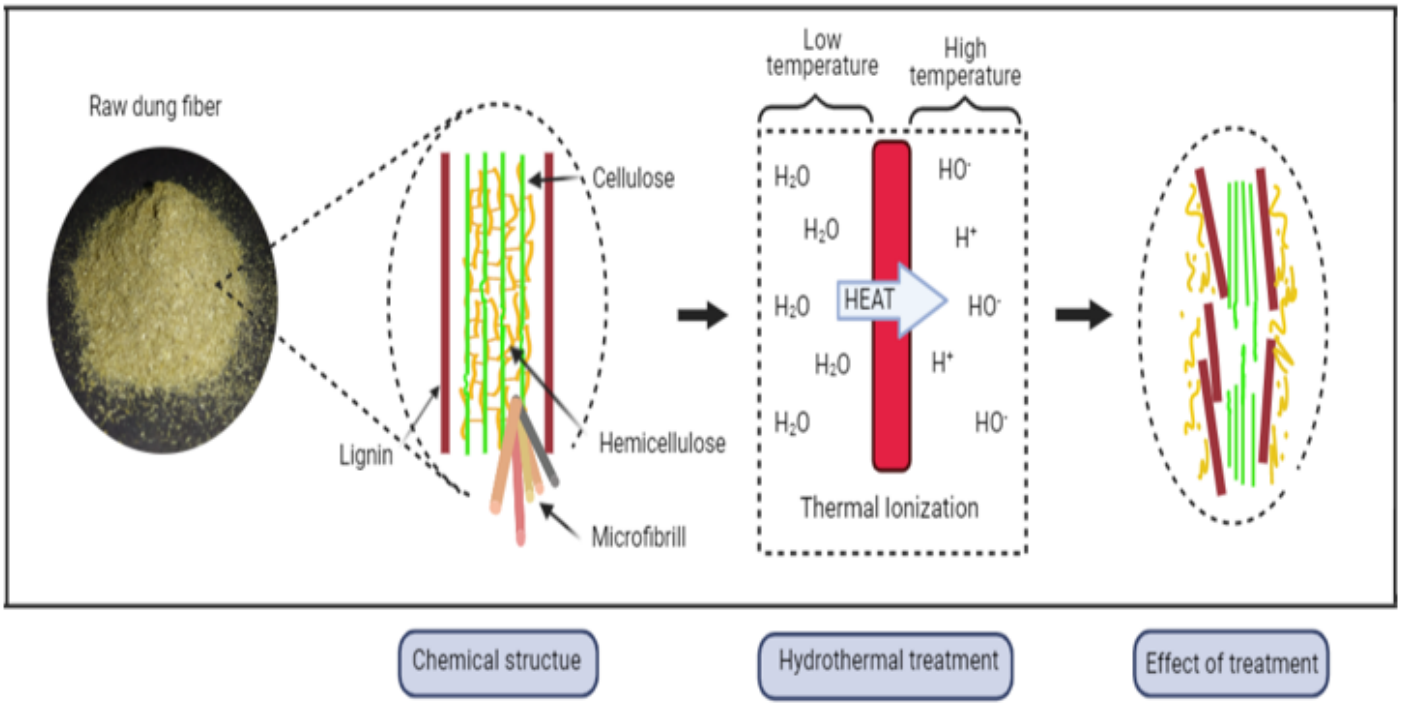

Figure 1

Schematic view of hydrothermal treatment of dung fibre material

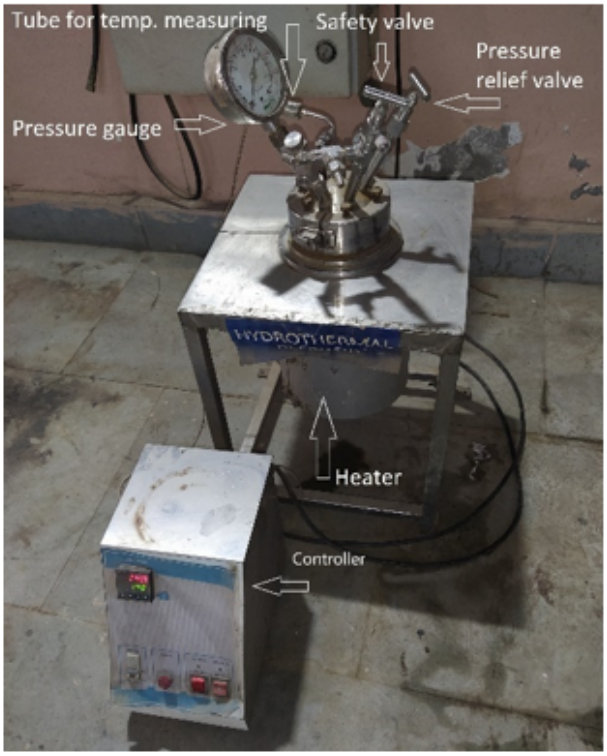

Figure 2

Hydrothermal reactor for the treatment of fibre

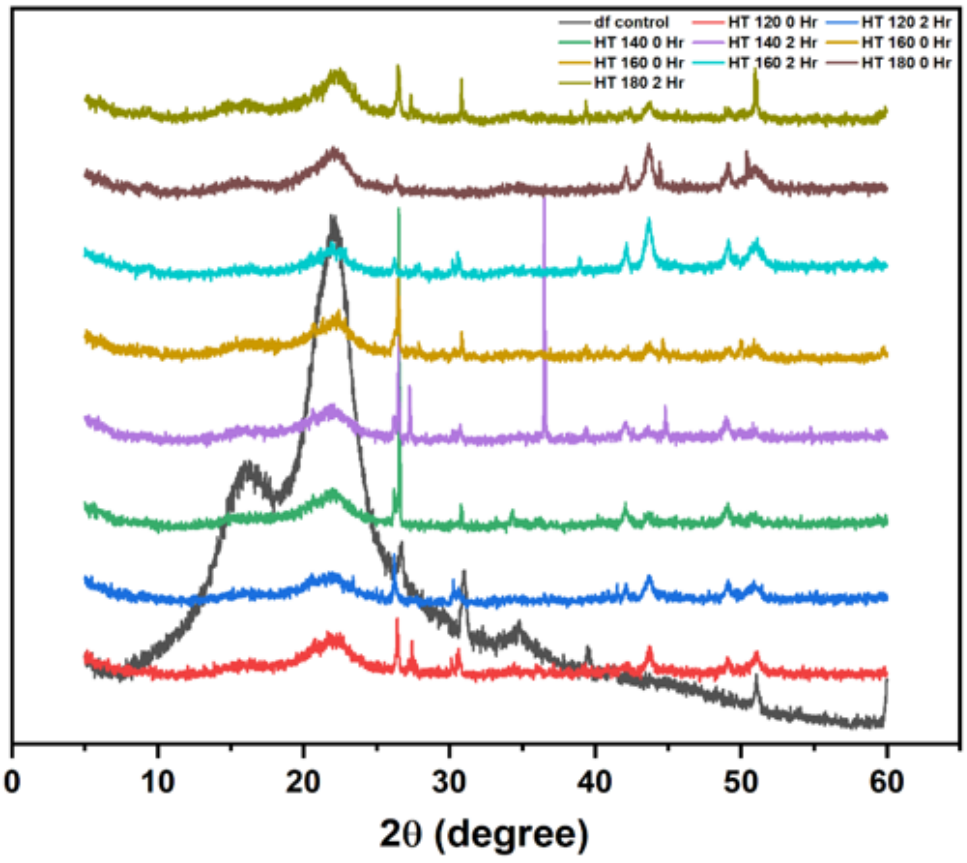

Figure 3

XRD patterns of dung fibre material (df) at different ageing times

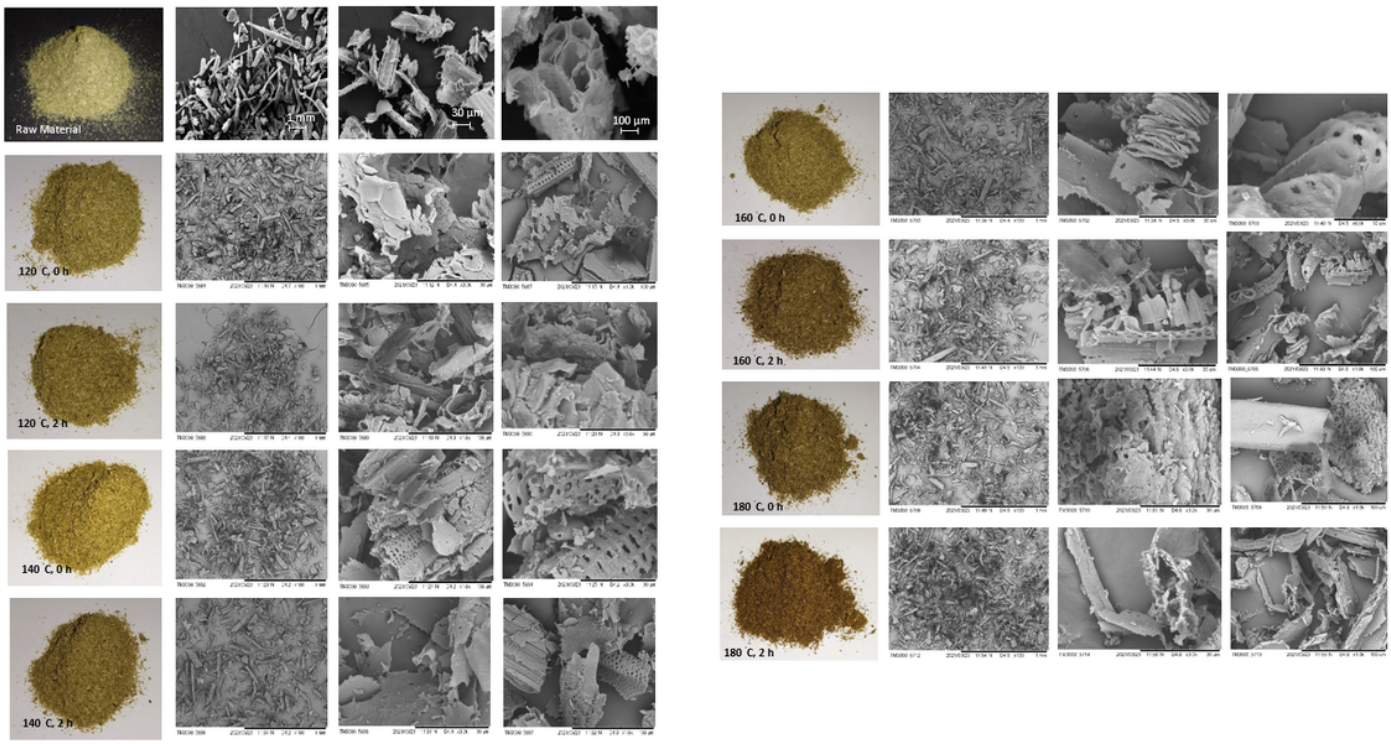

Figure 4

SEM images of the raw and the hydrothermally treated dung fibers with increasing temperatures and cooking period.

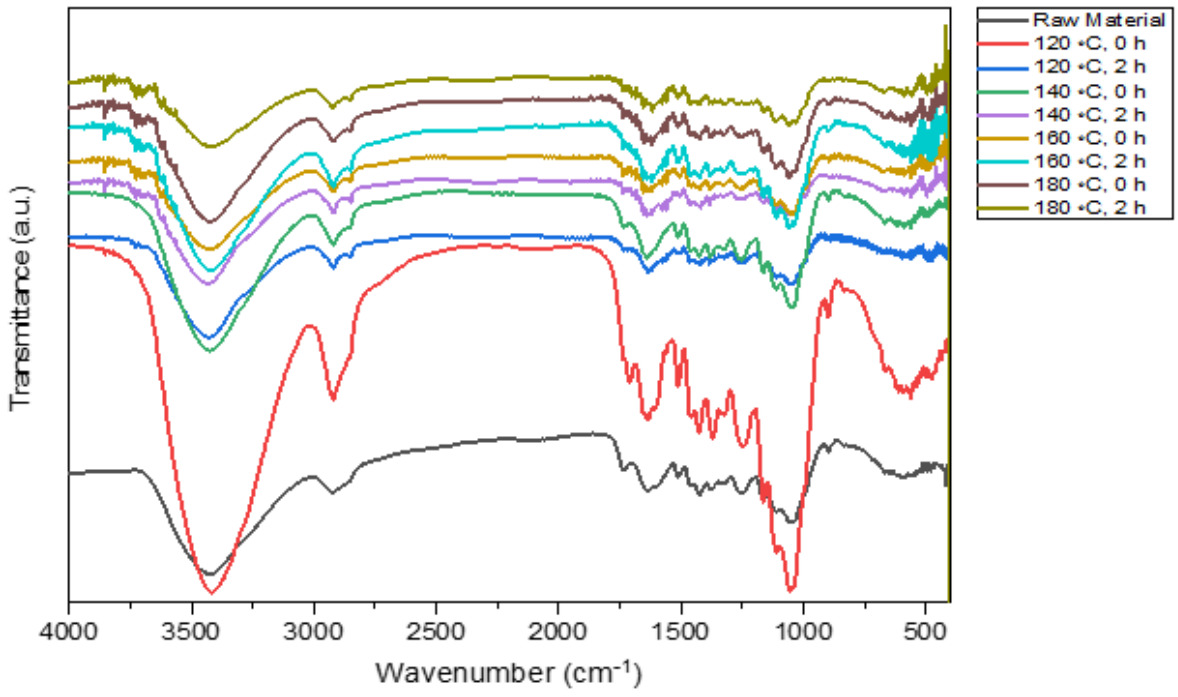

Figure 5

FTIR spectra of dung samples (untreated and hydrothermally treated)