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Research article

Valorization of cellulosic fiber derived from waste biomass of constructed wetland as a potential reinforcement in polymeric composites: A technological approach to achieve circular economy

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ABSTRACT

This study establishes the suitability of cellulosic fibers derived from *Canna indica* waste biomass for utilization as a reinforcement in natural fiber polymeric composites. The waste biomass was harvested from constructed wetlands engaged in the treatment of municipal wastewater from a gated community. The extracted *Canna indica* (CI) fibers were studied for their physicochemical, mechanical, structural, crystallographic, and thermal characteristics and proposed as a potential alternative to synthetic fiber. The CI fibers contained a relatively higher amount of cellulose (60 wt%) and a low wax fraction (0.5 wt%) – which is advantageous for its gainful utilization as a reinforcement. The CI fibers were thermally stable up to 237 °C and have an average fiber length, diameter, and density of 4.3 mm, 842 μm, and 0.75 g/cm^3, respectively. The mean maximum tensile strength and Young's modulus were found to be 113 ± 6.82 MPa and 0.8 ± 7.91 GPa, respectively. The nano-indentation test displayed the nano hardness and modulus as 0.3 ± 0.6 GPa and 1.62 ± 0.2 GPa, respectively. The crystallographic properties of CI fibers consisted of an 87.45% crystallinity index and 3.2 nm crystallite size. The morphological attributes of CI fibers showed rough surfaces and shallow cavities on the surfaces of the fibers suggesting the suitability for its utilization as a reinforcement. It is argued that this technological approach can potentially achieve circular economy through valorization of *Canna indica* biomass harvested from natural wastewater treatment plants.

1. Introduction

In recent years, remarkable emphasis has been laid on the sustainable management of municipal solid wastes (MSW) in general and waste biomass in particular for achieving UN sustainable development goals. The term 'biomass' typically refers to the biodegradable fraction of any products, wastes, and residues obtained from agricultural practices, animal husbandry, forestry, wood processing and fish farming industries, and biodegradable portions of industrial and municipal solid wastes (Union, 2009).

In this study, '*waste biomass*' refers to the biomass in the rural or urban community, which is typically not subjected to any value addition. In growing economies, researchers are making efforts for the valorization of waste biomass, primarily focusing on agricultural and forestry wastes for the production of energy (Nizami et al., 2017) (Santos-Ballardo et al., 2015), biochemicals (Rathore et al., 2016) (Naik et al., 2010), polymers (Henrique et al., 2013) (Kim et al., 2021), building materials (Barbieri et al., 2013) (Aouba et al., 2016) and carbon compounds (Nematian et al., 2021) (Mohamed et al., 2021). Currently, only a small fraction of waste biomass is being utilized to create value,

Abbreviations: Canna indica, CI; Waste Water Treatment, WWT; Constructed Wetlands, CWs; Natural Treatment Systems, NTSs; Domestic Wastewater, DWW; Wastewater, WW; Horizontal SubSurface Flow, HSSF; Indian Institute of Technology Bombay, IIT Bombay.

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while the majority remains unaddressed or is openly burned, causing significant environmental issues worldwide. (Tripathi et al., 2019) (Ates et al., 2020; Rana et al., 2021, 2022). Consequently, it is crucial to concentrate efforts towards valorizing biomass by implementing appropriate technology and establishing markets for utilizing environmentally-friendly products (Uppal et al., 2022; Beluns et al., 2021; Pappu et al., 2019a,b). This is essential for the transition from a petroleum-based economy to a bio-based economy and is the need of the hour (Pradhan et al., 2019) (Ravindra et al., 2019) (Platnieks et al., 2021; Zielińska et al., 2021).

Besides agricultural and forestry wastes, one of the emerging sources of waste biomass happens to be the biomass harvested from the domestic wastewater treatment (WWT) facilities that are based on the so-called natural treatment systems (NTSs), such as constructed wetlands, maturation ponds, and duckweed ponds (Arceivala and Asolekar, 2006) (Kumar and Asolekar, 2016) (Kumar et al., 2016) (Wintgens et al., 2016) (Sutar et al., 2021) (Lekshmi et al., 2020b) (Avellan et al., 2017) (Kasak et al., 2020). In the case of India, an estimated 15,000 MLD of rural domestic wastewater (DWW) could potentially be treated using constructed wetlands (CWs) in a decentralized manner – which happens to be nearly 40% of the wastewater (WW) generated in the rural communities (population approx. 900 million). An estimated 4000 MT/day could be the rate of generation of green biomass from treating nearly 15,000 MLD DWW using engineered CWs in rural India alone. The increasing interest of governments and municipalities in implementing decentralized NTSs for WWT due to various socio-economic and ecological benefits has raised several discussions regarding the valorization and scientific disposal of the waste vegetal biomass produced post-WWT (Arceivala and Asolekar, 2006) (Kumar et al., 2016) (Lekshmi et al., 2020a). The Indian Government and municipalities have employed more than a hundred facilities based on NTSs (Kumar et al., 2015). Interestingly, CW was found to be the widely accepted technology for WWT in urban, peri-urban, and rural regions of India due to low operation and maintenance cost, higher treatment efficiency, no skilled labor required, *etc*. (Sudarsan et al., 2015) (Rai et al., 2015) (Kumar et al., 2016) (Billore et al., 2001) (Mishra et al., 2018) (CPCB and DBT, 2019). Additionally, it is worth mentioning that due to techno-economic, social, and environmental benefits, CWs are a globally-accepted eco-centric WWT technology and have been employed in various parts of the world (Nivala et al., 2013) (Mthembu et al., 2013) (Everard et al., 2012) (Liu et al., 2009).

CWs are the replica of natural wetland systems that are typically designed and constructed to utilize the synergistic attenuation and buffering capacity of media, wetland biomass, and associated microbes for treatment of wastewaters (Vymazal, 2011a) (Brix, 1997). The above-ground vegetation needs to be periodically harvested to maintain the treatment efficiency and smooth functioning of CW systems (Vymazal, 2011b) (Datta et al., 2021) (Zhang et al., 2020). If the biomass is not harvested periodically, the resulting decomposing debris will reintroduce the nutrients removed *via* plant uptake and compromise removal efficiencies of organics and nutrients (Vymazal, 2011b) (Avellan et al., 2017).

Composting is one of the commonly explored applications (though not at a commercial scale) of harvested biomass generated from CWs and is mainly used by farming communities. It has been reported that mostly, waste biomass from CWs is typically used as fodder or soil conditioner (Licata et al., 2019) and the rest is generally discarded and left to decompose naturally, which leads to secondary environmental problems.

Waste biomass generated during WWT using CWs could be utilized as feedstock for various purposes such as the production of biogas (Solano et al., 2004) (Avellan et al., 2017), compost, fodder (Kivaisi, 2001), and recovery of nutrients (Masi, 2009). Apparently, in developing economies, no studies have been reported on the gainful utilization of waste biomass generated in response to WWT using NTSs such as CWs.

The increasing environmental awareness of the depleting forest resources, deforestation, soil erosion, nutrient leaching, and extreme changes in temperatures has raised severe concerns universally (Khan et al., 2022) (Uppal et al., 2022). Consequently, in recent years, the development of polymeric composites for their applications in various domains, such as construction, packaging, and automotive, has witnessed significant attention as the promising alternative to conventionally used wood materials (Thakur and Thakur, 2014) (Teuber et al., 2016). Polymeric composites constitute two components: polymeric matrix and reinforcing material (Medupin, 2013).

Utilization of waste biomass-derived reinforcing materials for the development of polymeric composites have been observed to have a significant upsurge in the past few years (Formela et al., 2022) (Mohanty et al., 2018). (Jiang et al., 2020) converted the industrial waste lignin into biochar and studied its potential application in reinforcing styrene-butadiene rubber as the substitute for carbon black. Carbocal - a solid residue obtained from the sugar beet industry as used a reinforcing agent for developing thermoplastic composite (Suffo et al., 2020). The strength characteristics of processed wheat straw-derived fibers were studied to assess their potential as an additive in the polypropylene-based composite (Panthapulakkal et al., 2006). (Madhoushi et al., 2014) studied the potential application of wood dust as a substitute for wood fibers for developing natural-fiber-reinforced composites (NFRCs). Also, extraction of reinforcing agents from municipal solid wastes (Ashori, 2008), agricultural residues and agro-industrial wastes (Ashori and Nourbakhsh, 2010) (Singha and Thakur, 2010), forestry wastes (Väisänen et al., 2016), wood-processing industry wastes (Miguez Suarez et al., 2003) and even garden waste (Viretto et al., 2021) is well-cited in the literature. Additionally, applications of various inorganic waste materials such as metallurgical slag (Barczewski et al., 2022), coal ash (Pappu, 2015), red mud (Suresh and Sudhakara, 2019), marble powder (Pappu et al., 2020), fly ash (Pappu and Thakur, 2017), (Kasar et al., 2020), jarosite (Pappu et al., 2019a,b), flue dust (Nimmagadda et al., 2014), blast furnace slag (Patnaik et al., 2019), waste rubber (Sathiyamoorthy et al., 2011) produced during industrial processing has also been used for the development of polymeric composites.

Integration of technological innovation with the circular economy principles is one of the best valorization practices to address the current challenges of waste biomass management generated from the treatment of DWW. It is envisaged that the cellulosic fibers extracted from the waste biomass will impart suitable engineering characteristics to the polymeric composites when used a reinforcement with respect to wood counterparts. The potential applications of the developed composites would be making furniture, panels, flooring tiles, false ceiling, or partitions used in schools, libraries, hospitals, homes, offices, and shopping complexes.

The present research established the suitability of extracted cellulosic fibers from waste biomass of constructed wetland for its utilization as reinforcement by investigating various physicochemical and mechanical characterizations. The structural, morphological, thermal, and chemical compositional attributes are also comprehensively studied through state-of-art-characterization techniques.

2. Materials and methods

The biomass (above-ground plants) used in the present study has been harvested from the pilot-scale horizontal subsurface flow constructed wetland (HSSF-CW) located at the Indian Institute of Technology Bombay (IIT Bombay). The HSSF-CW is planted with *Canna indica* (CI). It was thoroughly washed with tap water to remove any sticking soil and then chopped into pieces. The cleaned and chopped biomass was placed in a vessel filled with fresh water for a week at room temperature and pressure for retting. Subsequently, air-dried at 105 °C to remove all the moisture and fed to a shredder for fiber separation. The obtained coarse clusters of fiber and fragments of biomass are further subjected to a grinder to obtain short and fine pieces of fibers (Fig. 1 (a-

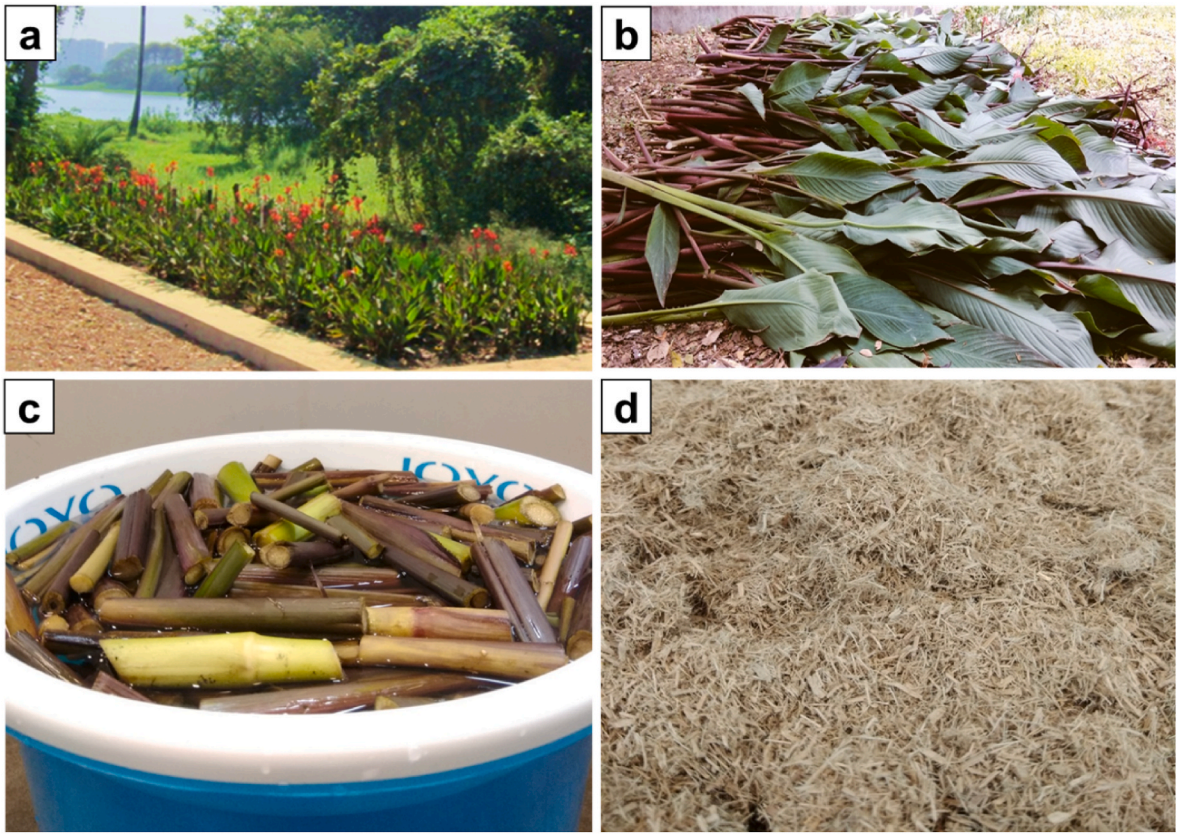

Fig. 1. Constructed wetland research station (a), harvested CI biomass (b), feedstock under retting process (c), and CI fibers (d).

d))

2.1. Physical characterizations of Canna indica fiber

a) Density

The density of the CI fibers was determined using ASTM standard D 3800–16 (Procedure -A, Archimedes method) (ASTM, 2010). Benzene having a density of 0.875 g/cm^3, was used as an immersion liquid.

b) Fiber Length

Image analysis software (Image-J) was used for measuring the fiber length. Initially, fibers were separately spread on dark-colored paper, and a digital image of each fiber was captured with a ruler in the calibration frame. The images taken were then loaded into the program for analysis. For length estimation, 100 fibers were measured.

c) Fiber Diameter

The diameter of the fibers was measured using an optical microscope (ZEISS Smartzoom5). The diameters were measured at four random places of 100 fibers.

d) Surface Roughness

The surface roughness parameters of CI fibers were measured using an alicona infinite focus confocal microscope (Bruker alicona, InfiniteFocusG6). The roughness parameters such as roughness kurtosis (R_{ku}), roughness skewness (R_{sk}), average roughness (R_a), root-mean-square roughness profile (R_q), maximum peak to valley height of roughness profile (R_t), and mean peak to valley height of roughness profile (R_z) were estimated as per ISO 4287:1997.

2.2. Chemical characterizations of Canna indica fiber

The moisture in CI fibers was estimated by adopting the ASTM standard D4442-16 (ASTM, 2007), while ash content was determined using ASTM standard E1755 – 01 (ASTM, 2001). Conrad's method (Conrad, 1944) was followed for estimating the wax content in the CI fibers, which involves the use of ethanol as an extraction agent for wax, resins, and fats, followed by the addition of chloroform for the extraction of wax from the alcoholic solution. To determine the hemicellulose proportion, 1 g of wax-free CI fibers mixed in 400 mL of 0.5 M NaOH solution was heated for 3 h, filtered, and washed till neutral pH, and then weighed post-oven-drying. Owing to the solubility of hemicellulose in an alkaline solution (Li et al., 2004) (Di Blasi et al., 1999), the difference in weight before and after NaOH treatment gives the hemicellulose content. Two-stage acid hydrolysis was performed to estimate the lignin proportion i (Lin et al., 2010) (Ayeni et al., 2013). A weighed amount of wax-free feedstock was mixed with 95% H_2SO_4 and left overnight with constant stirring. Thereafter, the mixture was diluted with 300 mL of distilled water, boiled for 3 h, and the obtained residue was washed and oven dried at 105 °C for 24 h. Post-acid-hydrolysis, the difference in weight reflected the acid-insoluble lignin content. Additionally, the absorbance of the acidic filtrate was measured at the wavelength of 320 nm to estimate the acid-soluble lignin content. The cellulose content estimation was carried out by assuming wax, hemicellulose, cellulose, lignin, and ash are the only constituents of the biomass.

2.3. Mechanical characterizations of Canna indica fiber

a) Single-Fiber Tensile Test

To determine the tensile characteristics of the CI fibers, single-fiber tensile tests were performed using a universal testing machine

(INSTRON. 5566 A) at a cross-head speed of 0.1 mm/min 100 single fibers were placed over the cardboard and held firmly at the cardboard ends using quick-setting polymeric adhesives. The test specimens had a gauge length of 20 mm.

b) Nano-indentation Test

The quasi-static nano-indentation test was conducted using diamond shaped Berkovich indenter with a radius of curvature <100 nm and a displacement resolution of <0.01 nm. During the test, 15 points were chosen with a maximum loading of 1.3 mN and loading and holding phases of 5 and 1s, respectively.

2.4. X-ray diffraction

X-ray powder diffraction (Miniflex, M/s. Rigaku Corporation, Japan) was performed to investigate the crystallinity and mineralogical composition of the CI fibers. The diffraction pattern was monitored in the 2θ range - 20–80 and step size - 0.02°. The crystallinity index of the cellulosic fiber was determined using the peak height method proposed by Segal and coworkers, where the empirical equation can be written as (Segal et al., 1959)

$$CI\ (\%) = \frac{I_{200} - I_{am}}{I_{200}} * 100 \quad (2)$$

Where CI is the crystallinity index, I_{200} is the diffraction peak of maximum intensity corresponding to 200 crystallographic planes at the 2θ angle between 22° and 23°; attributed to both crystalline and amorphous fractions, and I_{am} represents the peak intensity of amorphous fraction at the 2θ angle between 18° and 19°. In this study, I_{200} and I_{am} correspond to the diffraction peak at a 2θ angle = 22.37° and 18.3°, respectively. The crystallite size was calculated using Debye-Scherrer's equation and could be written as – (Haque et al., 2010) (Maache et al., 2017)

$$D_{hkl} = \frac{K\lambda}{\beta\ Cos(\theta)} \quad (3)$$

Where, D_{hkl} (nm) - average crystallite size, K - shape factor (0.9), λ - wavelength of CuKα radiation (0.15406 nm), β - full-width half maxima (FWHM) of diffraction peak under consideration, θ - diffraction angle (rad).

2.5. Field emission scanning electron microscopy and energy dispersive spectroscopy

The morphology of the feedstock was observed using FESEM (Nova NanoSEM 430, USA) operated at an electron-accelerating voltage of 300 kV. Samples were gold-coated for 90 s having a thickness of approximately 200 Å. The quantitative elemental study was also carried out using Energy Dispersive Spectroscopy (EDS), which comes along with the FESEM system.

2.6. Fourier transform infrared spectroscopy

To study the surface chemistry of the CI fibers, FTIR analysis was carried out. Infrared spectroscopy (Nicolet iS50, Thermo Scientific) was performed by the KBr pellet method in the region of 400–4000 cm^{-1}.1 mg of powdered biomass was mixed with 120 mg of high-purity KBr and pelletized.

2.7. Thermogravimetric analysis

The thermal behavior of CI fibers was analyzed using thermogravimetry analysis (Shimadzu DTG-60). Approximately 2 mg of powder biomass was placed in a platinum crucible and subjected to heating from 40 to 1000 °C at a heating rate of 5 °C/min under the nitrogen stream (N_2) having a flow rate of 100 mL/min. The change in mass during the ramping phase was estimated.

2.8. X-ray photoelectron spectroscopy

X-ray photoelectron spectroscopy (Kratos Analytical, AXIS Supra) having monochromatic Al-Kα (1486.7 eV) as the X-ray source was performed to determine the surface elemental concentrations of CI fibers. For data acquisition, 10 scans from a single point were noted between 1350 and 10 eV. The CI fiber was sputtered with Ar gas prior to analysis.

3. Results and discussion

3.1. Lignocellulosic compositional and surface morphological suitability

The compositional attributes of lignocellulosic fibers are of great significance as it governs their suitability (mechanical characteristics, biodegradability, and structural integrity) for its application as a reinforcing element in developing NFRCs (Komuraiah et al., 2014). In addition, surface morphology provides information about the surface texture, which influences its ability to reinforce and resist fiber-pull out during NFRCs failure. Therefore, these characteristics are studied for CI fibers and are discussed below in detail. The chemical composition of CI fiber is presented on a dry weight basis in Table 1, along with other natural fiber.

The amount of wax was limited to 0.5 wt %, which is beneficial for the use of CI fiber as a reinforcement as the higher value will lead to smooth morphology and weaken the interfacial bonding between fiber and matrix. The cellulose content was found to be 60% which is relatively higher than *Corypha taliera fruit fiber* (55.1%) (Tamanna et al., 2021), *Ferula communis* (53.3%) (Seki et al., 2013) Cissus quadrangularis stem (27%) (Indran and Raj, 2015), *Arundo donax* (43.2%) (Fiore et al., 2014a) thus making CI fiber a potential reinforcing element for developing NFRCs. Cellulose is known to impart stiffness, strength, structural stability, and resistance to hydrolysis to natural fibers (Sajith et al., 2017). Therefore, among other components, cellulose is considered the major framework constituent for its application in composite fabrication (Shesan et al., 2019). The lower amount of hemicellulose (10.68%) could have benefits for its use as a reinforcement as higher hemicellulose causes poor tensile strength, higher thermal degradation, and hydrophilicity (Komuraiah et al., 2014) (Park et al., 2008).

The lignin content was found to be relatively higher (20.52%) than other reported natural fibers, which could be an added advantage as lignin acts as an encrusting material for the fiber constituents and provides structural rigidity, water resistance, and resistance against microbial attack (Weng et al., 2020) (Zwawi, 2021). Approximately 8.9% ash content was noted. Ash primarily consists of SiO_2, and reportedly the presence of silica can enhance the elastic modulus and hardness of natural fibers; the higher ash content is also correlated to the improved lateral bonding of fibers (Wu et al., 2010).

The values of tensile strength and cellulose for various natural fibers are shown in Fig. 2. The flax and banana fibers showed the highest tensile strength even though their cellulose content was lower than cotton and sisal fiber, respectively. A similar trend was noted for other fibers too. It is true that, in natural fibers, the cellulosic microfibrils impart tensile strength (Weng et al., 2020). However, the cellulose proportion and its type and geometrical conditions primarily control the tensile properties of natural fibers (Das et al., 2019) (Bledzki and Gassan, 1999). Here, the presented tensile strength and cellulose content is the average value calculated from the given range in the published literature.

The surface morphology of the CI fiber was investigated using FESEM and has been presented in Fig. 3 (a-e). The longitudinal view of the CI fiber (Fig. 3-(a)) showed rough surfaces and shallow cavities on the fiber surface. For developing NFRCs, the rough texture of natural fibers is

Table 1
Comparison of physicochemical and mechanical properties of CI fiber and other lignocellulosic fibers.

Lignocellulosic Fiber	Density (g/cc)	Diameter (μm)	σ (MPa)	E (GPa)	Nano hardness (GPa)	Wax (%)	Hemi cellulose (%)	Cellulose (%)	Lignin (%)	Ash (%)	Moisture* (%)	Reference
Canna indica	0.75	842	113 ± 6.82	0.8 ± 7.91	0.3 ± 0.6	0.5	11	60	20	9	8	Present study
Corypha taliera fruit	0.86	131	53.55	451.81	–	–	21.78	55.1	17.6	–	7.1	Tamanna et al. (2021)
Luffa vine	0.20–0.60	–	–	–	0.40–0.58		15.1 ± 0.8	54.6 ± 0.4	–	10.5 ± 0.5	–	Weng et al. (2020)
F. communis	1.24	90–300	475.6 ± 15.7	52.7 ± 3.7	–	–	8.5	53.3	1.4	7.0	24.8	Seki et al. (2013)
Cissus quadrangularis stem	1.22	770–870	4203 ± 1276	131 ± 37	–	0.18	7.96	82.73	11.27	–	6.6	Indran and Raj (2015)
Arundo donax L	1.168	–	248	9.4	–	–	20.5	43.2	17.2	1.9	–	Fiore et al. (2014a)
Coconut coir	1.15	100–450	500	2.5	–	–	12.1	44.2	32.8	–	11.36	Sathishkumar et al. (2013)
Althaea officinalis L.	1.18	–	415.2	65.4	–	–	13.5	44.6	2.7	2.3	–	Sarikanat et al. (2014)
Prosopis juliflora	–	–	558 ± 13.4	–	–	0.61	16.14	61.65	17.11	5.2	9.48	Saravanakumar et al. (2014)
Kenaf	1.31	65–71	427–519	23.1–27.1	–	0.8	8–13	45–57	21.5	–	6–12	Indran et al. (2014)
Sansevieria cylindrica	0.91	6–30	656.83 ± 55	5.81 ± 2.8	–	0.09	10.13	79.7	3.8	–	6.08	Sreenivasan et al. (2011)
Aerial roots of banyan tree	1.23	0.09–0.14	19.37 ± 7.72	1.8 ± 0.40	–	0.81	13.46	67.32	15.62	3.96	10.21	Ganapathy et al. (2019)
Areca palm leaf stalk	1.09 ± 0.017	285–330	334.66 ± 21.46	7.64 ± 1.13	–	0.71	18.34	57.49	7.26	1.43	9.35	Shanmugasundaram et al. (2018)
Thespesia populnea barks	1.41	10–40	557.82 ± 56.29	20.57 ± 4.46	–	0.76	12.64	70.12	16.34	1.80	10.83	Kathirselvam et al. (2019)
Acacia leucophloea	1.38	168.5	317–1608	8.41–69.61	–	0.55	13.6	68.09	17.73	0.08	8.83	Arthanarieswaran et al. (2015)
Ipomoea staphylina	1.4	–	173–658	–	–	1.51	13.6	72.76	19.56	1.40	8.28	Santhanam et al. (2016)
Furcraea foetida	0.778	12.8	594.52 ± 48	6.15 ± 1.4	–	0.24	11.46	68.35	12.32	6.53	5.43	Manimaran et al. (2018)
Ficus religiosa	1.246	25.62	433.32 ± 44	5.42 ± 2.6	–	0.72	13.86	55.58	10.13	4.86	9.33	Moshi et al. (2020)
Sisal	1.5	50–300	511–635	9–22	–	2.0	10–14.2	60–78	8–14	–	10–22	Indran and Raj (2015)
Conium maculatum	–	14.12	327.89 ± 67.41	15.77 ± 3.15	–	–	32.2	49.5	8.6	–	–	Kılınç et al. (2018)
Perotis indica	0.785	–	317–1.608	8.41–69.61	–	0.32	15.7	68.4	8.35	4.32	9.54	Prithiviraj et al. (2016)
Heteropogon Contortus	0.602	–	476 ± 11.6	48 ± 2.8	–	0.22	19.34	64.87	13.56	–	7.4	Hyness et al. (2018)
Epipremnum aureum stem	0.654	105–150	317–810	8.41–69.61	–	0.37	13.42	66.34	14.01	4.61	7.41	Maheshwaran et al. (2018)

Note: The chemical compositions reported above are on a dry weight basis except moisture.

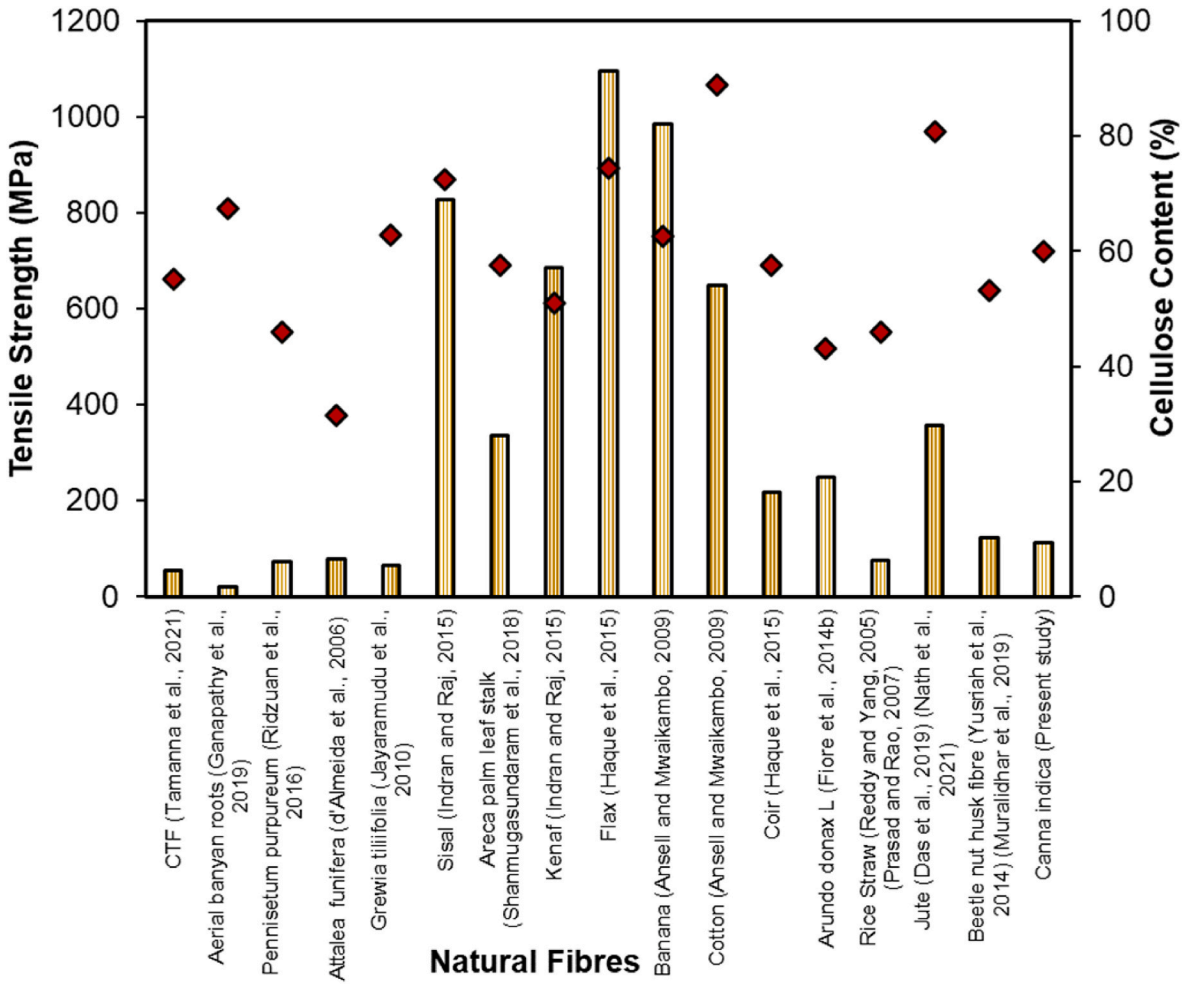

Fig. 2. Variation of tensile strength and cellulose content for various natural fibers.

desirable as the surface roughness and cavities present augments the mechanical interlocking between natural fiber and polymeric matrix, consequently enhancing the fiber-matrix interfacial stability (Sepe et al., 2018). Such morphological characteristics also improve the thermal characteristics of the composite materials (Dalmis et al., 2020).

In (Fig. 3-b) it can be seen that CI fiber, like other lignocellulosic fibers such as Arundo fiber (Fiore et al., 2014a) and banana fibers (Kambli et al., 2016), is composed of several elongated individual fiber cells, commonly referred to as microfibrils bonded together along the direction of their length by non-cellulosic compounds such as pectin, hemicellulose, and lignin. The fiber cells showed a compact arrangement having a diameter ranging between 3 and 8 μm. The cross-sectional image of the CI fiber strand (Fig. 3-c) was also captured to have a better visualization of fiber structure, and it was observed that the fiber strand consisted of pores and numerous nearly spherical-shaped microfibrils diameters ranging from 2 to 11 μm arranged together to form an almost cylindrical structure. Similar observations were reported for date palm rachis fibers by (Boumediri et al., 2019). At higher magnification (Fig. 3-**d**), the morphological attributes of CI fiber exhibited a mat-like structure consisting of cellulose microfibrils and other non-cellulosic materials. Further (Fig. 3-e), shows the impurities (wax, lignin), a typical characteristic of plant fibers, on the CI fiber surface. The impurities on the fiber surface can be removed through various chemical treatments and are one of the commonly applied techniques to improve the fiber-matrix bonding in NFRCs.

The qualitative and quantitative analysis of the surface elemental composition of CI fiber was performed using EDS. The findings suggested that carbon (59.63 wt%) and oxygen (35.33%) constitute the major elements phase in CI, along with the presence of potassium (K), calcium (Ca), magnesium (Mg), copper (Cu), iron (Fe), chlorine (Cl) and silica (Si). In summary, the CI fiber possess relatively higher content of cellulose and therefore may prove to be a superior reinforcement in developing NFRCs.

3.2. *Mechanical characteristics*

The mechanical characteristics of the CI fiber were assessed in terms of the single fiber tensile test and nano-indentation test and are shown in Table 1.

a) Single Fiber Tensile Test

The tensile characteristics of CI fibers were determined by performing the single-fiber tensile test for 100 fibers, and the tensile strength and modulus were reported. The CI fiber exhibited a mean maximum tensile strength of 113 ± 6.82 MPa, which is superior to reported natural fibers, including aerial roots of banyan (19.37 MPa) (Ganapathy et al., 2019), Pennisetum purpureum (73 MPa) (Ridzuan et al., 2016), and Napier grass (75 MPa) (Reddy et al., 2009). The tensile modulus was found to be 0.8 ± 7.91 GPa which indicates the stiffer nature of CI fiber. During the tensile test, CI fiber displayed brittle nature with nearly zero plastic deformation. The standard deviations of the tensile parameters are moderately higher, particularly in natural fibers. It is apparent due to several factors, including plant age, environmental

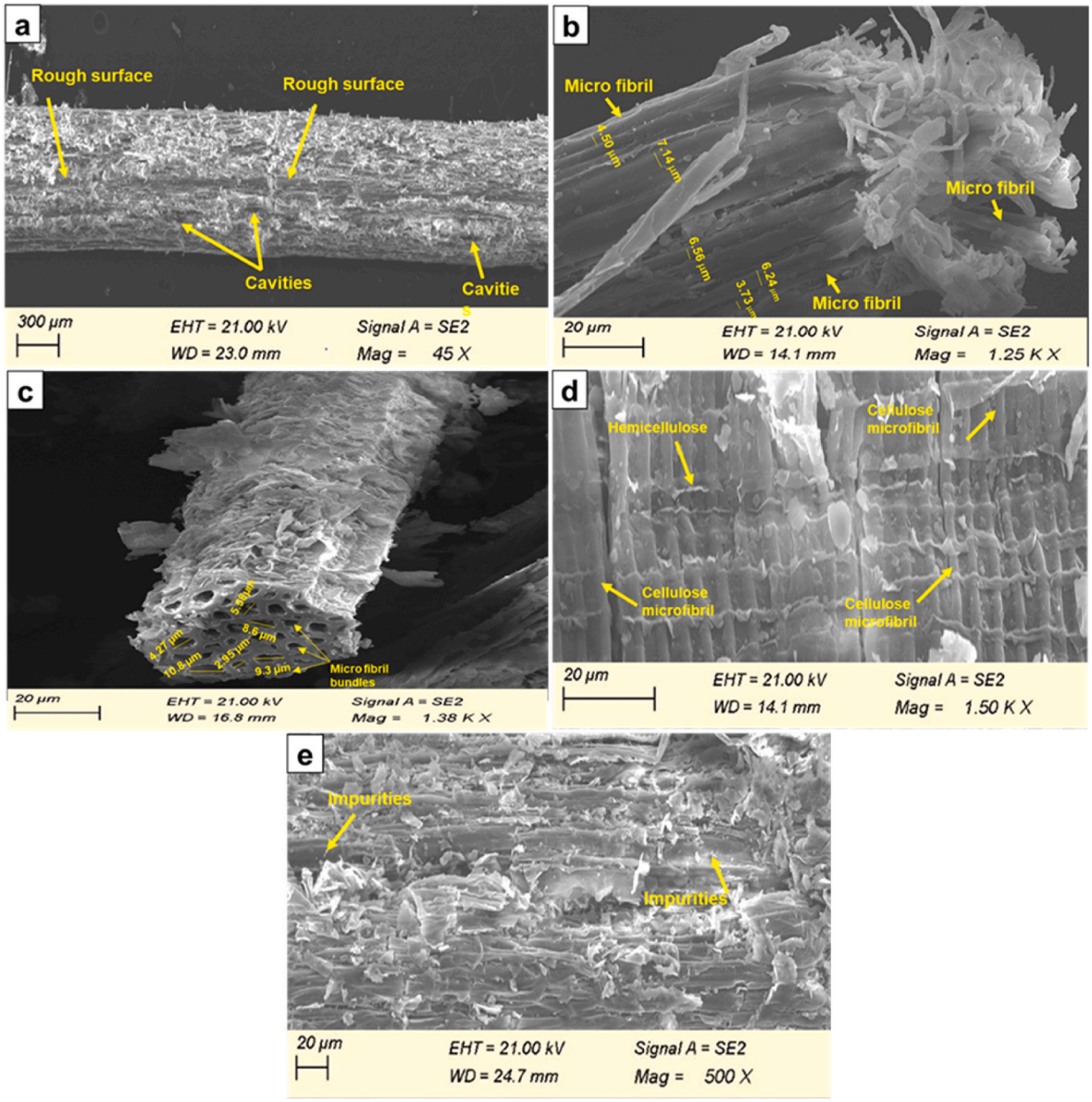

Fig. 3. FESEM micrographs of CI fiber at different magnifications (a–e).

conditions during growth, surface imperfections, and assumptions about circular cross-section and test conditions (Fiore et al., 2014b).

b) Nano-indentation Test

For estimating the micro-mechanical properties of CI fibers, epoxy resin was used as the embedding medium due to its relatively low modulus and negligible effect on the nano-indentation test data of natural fibers (Tze et al., 2007). The nano hardness and nano modulus of CI fibers were found to be 0.3 ± 0.6 GPa and 1.62 ± 0.2 GPa, respectively.

3.3. Favorable physical and topographicalcharacteristics

The physical characteristics of CI fiber was studied in terms of fiber length, diameter, and surface roughness. The average fiber length was measured to be 4.5 mm, and the mean diameter is estimated to be 842 µm **(Figure SM 1 (a))** (**Supplementary Material**). The density of CI fiber was estimated to be 0.75 g/cm^{3,} which is much lower than carbon fibers (1.75–1.93 g/cm^{3}) (Gulgunje et al., 2015); thus making it a promising candidate for developing lightweight NFRCs. Table 1 shows the physical properties of CI and other lignocellulosic fibers. As suggested by (Sreenivasan et al., 2011), the low-density and porous natural fibers may be suitable for fabricating thermal and acoustic insulation panels for various applications – which seems to be the case for CI fibers.

Further, the topographic attributes of CI fiber are presented in the form of 3-D surface texture along with the roughness profile parameters (**Figure SM 1 (b)**) (**Supplementary Material**). The undulations observed in the 3-D image point toward the irregularities (peak and valley) on the fiber surface. The average roughness (R_a) value was found to be 3.27 µm, which is relatively higher than that of *Cyperus pangorei* (0.625 µm) (Mayandi et al., 2016) and *Chloris barbata* (0.894 *10^{-3} µm) (Balasundar et al., 2018) and comparable to palm rachis fiber (3.4 µm) (Boumediri et al., 2019).

The higher value of average roughness shows the rough texture of the fiber surface and the presence of lower amounts of impurities (wax, pectin) which will improve the interfacial adhesion between the fiber and polymeric matrix in NFRCs (Arthanarieswaran et al., 2015). The other important parameters determined in the present study are roughness kurtosis (R_{ku}) and roughness skewness (R_{sk}). The R_{ku} determines the surface types, while the R_{sk} examines the symmetrical

profiling of surface variations along the centerline (Manimaran et al., 2018). A value of R_{ku} >3 signifies the spiky surface (Csanády et al., 2019). For CI fiber, the R_{ku} value was obtained as 7.49, thus referring to spiky surface structures. Moreover, the R_{sk} was noted to be −0.73.

Here, the negative sign shows the porous characteristics of the lignocellulosic fiber, i.e., the fiber surface consists of valleys, pits, and cavities (Liang et al., 2018), (Senthamaraikannan et al., 2016). The observations made during surface profiling are in coherence with the FESEM analysis discussed in the above section. Additionally, the higher value of Rz (24.19 μm), Rt (40.76 μm), and Rq (5.52 μm) obtained also supports the rough texture of the CI surface.

3.4. Crystallographic and thermal characteristics

Crystallinity serves as an important parameter to characterize lignocellulosic fibers when it comes to their use as a potential reinforcement, as it significantly influences the toughness and mechanical characteristics of the fiber (Petroudy, 2017) and thus has been determined in this study. Generally, an increase in crystallinity is accompanied by an increase in stiffness, Young's modulus, dimensional stability, heat resistance, and strength (Petroudy, 2017). In addition, the manufacturing processes of NFRCs involve high temperatures; therefore, the study of the thermal stability of CI fiber is imperative to determine its onset degradation temperature.

The XRD spectrum of CI fiber (Fig. 4) showed characteristic peaks of naturally occurring polymorph of cellulose consisting of parallel strands, *i.e.*, Cellulose (I) at 15.0°, 22.37°, 24.11° and 35.3° corresponding to the (1–10), (200), (002), (004) crystallographic planes (Senthamaraikannan et al., 2016), (Dalmis et al., 2020). The sharp peaks of the cellulose I indicate the orderly arrangement of the cellulose crystallites along the fiber axis. Furthermore, the diffraction peaks that appeared at 30.04 and 38.18 can be related to the presence of KCl in the CI plant structure. The presence of sylvite (KCl) has also been reported by (Cui et al., 2017).

The crystallinity index of the CI fiber was calculated to be 87.45% which is higher than Lygeum spartum L. (46.1%) (Belouadah et al., 2015), Cissus quadrangularis stem (47.15%) (Indran and Raj, 2015), Althaea officinalis L. (68%) (Sarikanat et al., 2014), Calotropis gigantea fruit fiber (36%) (Narayanasamy et al., 2020); comparable to hemp fibers (88%) (Sreenivasan et al., 2011). A higher crystallinity index reflects the long-range ordered arrangement of cellulose crystallites. The crystallite size (D) calculated using Debye-Scherrer's formula for the crystallographic plane (200) was 3.2 nm. The crystallite size reported for some other lignocellulose fibers is flax (2.8 nm), cotton (5.5 nm), and corn stalk (3.8 nm) (Indran et al., 2014).

The TGA and DTG curve of CI fiber exhibited four staged degradation profiles. The thermal behavior of CI fiber is shown in Fig. 5. The first stage of degradation occurred between room temperature to 210 °C with a mass loss of 8.77% attributed to the loss of physically adsorbed water, low-molecular-weight compounds (extractives), and chemically bounded water to structural components of lignocellulosic fibers (Fiore et al., 2014a). The second stage (9.59% mass loss) of the mass-loss profile observed between 210 and 260 °C, with onset degradation temperature (T_{onset} [°C]) at 237 °C, can be correlated with the thermal depolymerization of hemicellulose, glycosidic linkages in cellulose structure, and pectin (Saravanakumar et al., 2014).

A maximum weight loss of 35.01% was noticed in the temperature range of 260–400 °C, corresponding to the thermal degradation of cellulose. The thermal decomposition of cellulose produces anhydrocellulose, levoglucosan, water molecules, carbon dioxide, and alkanes (Elanthikkal et al., 2010) (Mohan et al., 2006). In the DTG curve, a prominent peak appeared at 301.4 °C, which indicates the decomposition of cellulose I and α-cellulose (Manimaran et al., 2018). The further mass loss (31.11%) occurring in a wide temperature range from 400 to 900 °C could be linked to the lignin degradation, and the same has also been reflected in the DTG curve at 444.3 °C and 608.4 °C. The thermal decomposition of lignin occurs at a relatively lower rate and over a wide range of temperatures ranging from ambient to 900 °C due to its complex three-dimensional aromatic structure (Asim et al., 2020). Evidently, CI fibers can be utilized for NFRCs manufacturing with processing temperatures below 237 °C which suits its processing with thermoplastic polymeric matrices. The obtained value is comparable with the ones reported in other studies, as shown in Table SM 1 (Supplementary Material).

3.5. Significance of surface functionality and chemical composition

The surface functional groups and surface chemical composition of the CI fiber were investigated using FTIR and XPS in order to comprehend their surface polarity and chemistry. These factors are crucial in determining how CI fiber interacts with polymeric matrices during the development of polymeric composites.

The qualitative investigation of the surface chemistry of CI fiber was carried out using FTIR analysis (Fig. 6). The FTIR spectrum of CI fiber displayed transmittance bands associated with polysaccharides, lignin, and glycosidic linkages in the CI fiber. A broad band at 3345 cm^{-1} relates to the O–H stretching vibrations of –OH groups present in organic and inorganic constituents of CI fiber and water molecules. The peaks located at 2926 cm^{-1} and 2850 cm^{-1} can be assigned to the asymmetric and symmetric stretching vibrations of aliphatic C–H bonds occurring in hemicellulose and cellulose (Siva et al., 2020), while a shoulder at

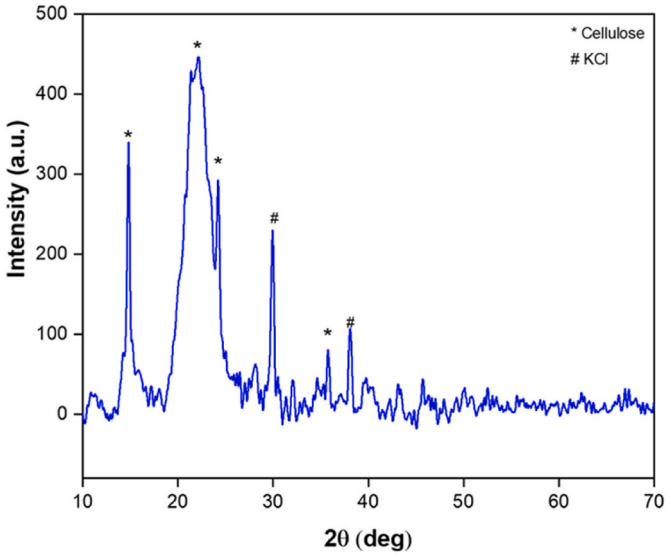

Fig. 4. XRD spectrum of CI fiber.

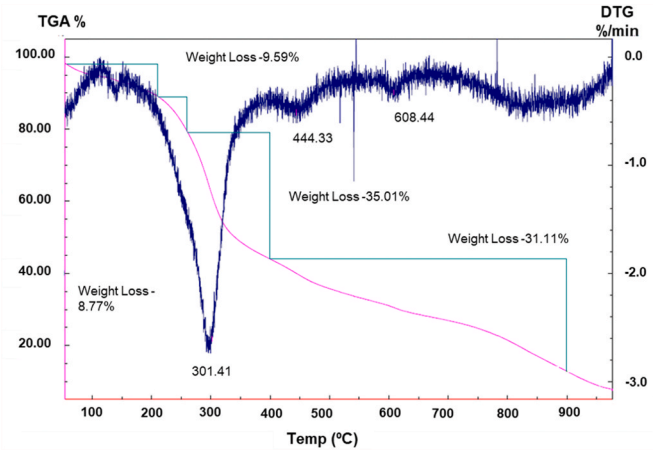

Fig. 5. TGA-DTG of CI fiber.

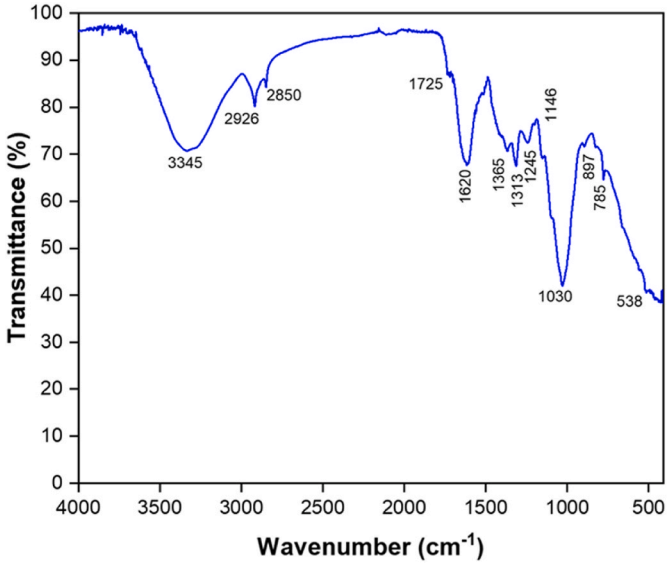

Fig. 6. FTIR spectrum of CI fiber.

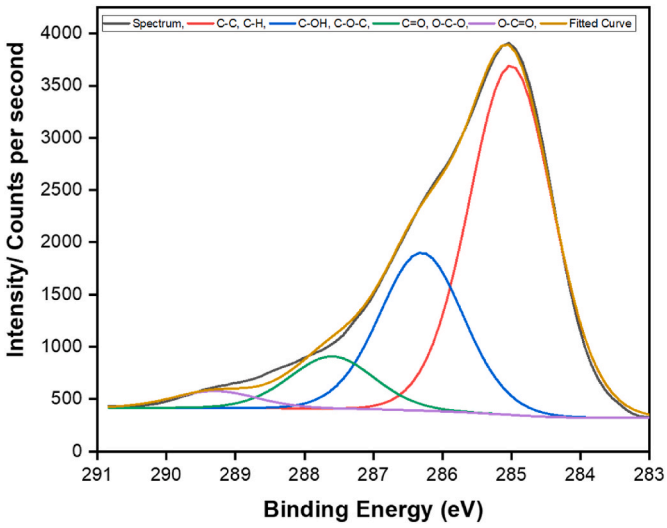

Fig. 7. XPS spectrum of CI fiber.

wavenumber 1725 cm^{-1} designates the stretching vibrations of carbonyl (C═O) groups in hemicellulose (Liu et al., 2016). Here, the appearance of the shoulder peak indicates the minor content of hemicellulose in CI fiber which is advantageous as the higher hemicellulose content adversely affects the mechanical strength of the natural fiber (Chen et al., 2015).

The sharp peak at 1620 cm^{-1} is correlated with the C═O and C═C stretching vibration of the lignin aromatic rings (Sathishkumar et al., 2013); the appearance of prominent lignin peaks is favorable for the use of CI fiber as a potential reinforcement for NFRCs as lignin helps in preventing the fungal and termite attack and imparts rigidity to the lignocellulosic fiber. The bending vibrations of C–H groups in cellulose and acyl groups (C═O) in aromatic rings were also noticed as a small peak at 1365 cm^{-1} and 1313 $cm^{-1,}$ respectively (Rajeshkumar et al., 2021). The transmittance band positioned at 1245, 1146, and 1030 cm^{-1} is due to the C–O stretching vibration of the acetyl group in lignin (Tawakkal et al., 2016), asymmetrical stretching of ether groups (C–O–C) in cellulose and hemicellulose (Moussaoui et al., 2021), and stretching of C–O and C–H functionalities in cellulose (Tamanna et al., 2021). The peak observed at 897 cm^{-1} points to β-glycosidic linkages in cellulose (Babu et al., 2019). The peaks in the region of 540–785 cm^{-1} correspond to the occurrence of chloride-based compounds.

The relative surface composition of CI fibers as determined using XPS was C (74.1%), O (21.49%), Ca (0.6%), P (1.4%), Si (1.0%), and Mg (1.0%). The estimated O/C ratio for CI fiber was found to be 0.29. As reported by (Laine et al., 1994), among the constituents of natural fibers, carbohydrates, and pectin possess the highest O/C ratio (0.83), followed by lignin (0.35) and extractives. Since the O/C ratio of CI fiber is lower than 0.83 therefore, it can be inferred that the CI fiber surface possesses higher content of aliphatic and aromatic carbon due to rich lignin content and is relatively less polar. Thus, CI fiber can provide good interfacial compatibility with non-polar polymers and could be utilized as a promising reinforcement for developing polymeric composites.

Furthermore, for quantitative assessment of functional groups present in CI fiber, the C1s peak was deconvoluted. Fig. 7 shows the deconvoluted C1s spectra. It was observed that the CI fiber consisted of diverse functionalities such as C–C/C–H, C–OH/C–O–C, C═O/O–C–O, and O–C═O with a relative proportion of 60.61, 27.39, 9.08 and 2.93% respectively (Table SM 2 (Supplementary Material).

3.6. Technological advantages and circular economy benefits

The synthetic fiber-reinforced polymeric composites have been widely used lately in mechanical and structural industrial applications. Owing to their undesirable environmental impacts, lignocellulosic biomass is being explored to develop commercially viable NFRCs. Unfortunately, the cost of collection and transportation of the lignocellulosic biomass from agriculture and forests is expensive and labour intensive. This study aims to break this impasse by obtaining high-quality lignocellulosic feedstock from eco-centric natural wastewater treatment technology based on constructed wetlands that can potentially treat domestic and industrial wastewaters from urban and peri-urban communities including clusters of rural communities. This study highlights the potential of utilizing biomass from such wastewater treatment facilities, especially those in large urban municipalities and metro cities as a feedstock for large-scale NFRC production plants.

On a closing note, to implement this technology commercially, the research is progressing on three fronts: (1) assess the opportunities and challenges associated with commercial exploitation, (2) develop a variety of NFRCs using harvested waste biomass of CWs-based wastewater treatment technologies and (3) aim to achieve UN sustainable development goals and circular economy.

4. Conclusions

In this research, a technological approach for the valorization of waste biomass produced in response to domestic WWT by CW has been proposed, which will subsequently enhance the sustainability of CW technology. Various attributes of CI fiber were studied to evaluate its potential to be used as a reinforcing element for polymeric composites. The higher cellulose fraction (60 wt%) and lower wax content (0.5 wt%) ensure better interfacial interaction of CI fiber with polymeric matrix during composite manufacturing. Due to low density (0.75 g/cm^3) compared to synthetic fibers, the CI fibers can be utilized to produce lightweight composite materials. Morphological features showed a typical structure of lignocellulosic fiber with a rough texture and shallow cavities, which is in coherence with the surface roughness measurement. The rough texture and surface cavities will promote mechanical interlocking between filler and matrix, thus advantageous for NFRC development. Single fiber test exhibited comparable mean maximum tensile strength (113 ± 6.82 MPa) and Young's modulus (0.8 ± 7.91 GPa) with that of reported lignocellulosic fibers. While nano hardness and nano modulus of CI fibers were obtained as 0.3 ± 0.6 GPa and 1.62 ± 0.2 GPa, respectively. FTIR and XPS analysis characterized

the surface functionalities, which showed diverse functional groups associated with the lignocellulosic components of CI fiber. The CI fibers were thermally stable up to 237 °C, which is higher than the degradation temperatures of thermoplastic matrices indicating that the CI fiber can be potentially used as the reinforcing element for developing thermoplastic-reinforced composites.

One of the major roadblocks while commercialising NFRCs happens to be the costs associated with collection and transportation of biomass and connected labour. In the context of propagation of the natural treatment systems and nature-based solutions being favoured lately in India; it is argued here that wastewater treatment facilities in large urban municipalities and metro cities will provide access to huge quantities of waste biomass and thereby provide opportunities for commercialising NFRCs. This study establishes the utility of waste-derived CI fibers for potential use to develop NFRCs for multifunctional applications. It is hoped that the technological approach proposed in this research will facilitate circular economy by presenting sustainable alternatives to synthetic fibers as well as enhance the propagation of eco-centric technologies for wastewater treatment.

Credit author statement

Shruti Sharma: Conceptualization, Methodology, Investigation, Visualization, Data curation, Writing – original draft; Shyam R. Asolekar, P Asokan: Methodology, Visualization; Supervision; Investigation, Writing – review & editing; Resources, Funding acquisition; Vijay Kumar Thakur: Writing – review & editing; Resources, Funding acquisition; Supervision.

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Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jenvman.2023.117850.

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