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ORIGINAL PAPER

Recycling Baby Diaper Waste into Cellulose and Nanocellulose

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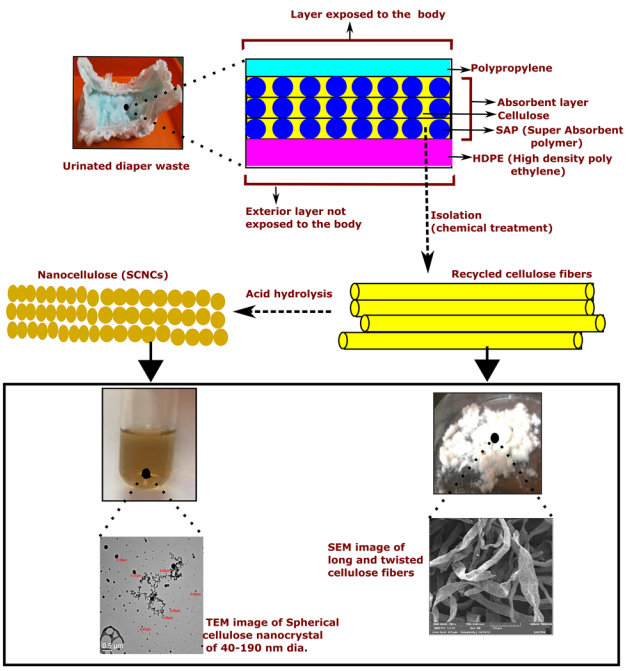
Abstract

The present work reports the baby diaper wastes to recycle into cellulose and nanocellulose. Cellulose was extracted by chlorine-free approach, using acetic and nitric acids. Approximately 25.13 ± 1.02% w/w cellulose was extracted from the used baby diapers. FE-SEM and TEM analysed the morphology of extracted cellulose and nano cellulose. Different carbon atoms and functional groups of the extracted cellulose were identified by CP/MAS ^{13}C NMR and FTIR spectroscopy respectively. Extracted cellulose exhibited a crystallinity of 65.16% as observed from the XRD analysis. A horizontal pattern of weight loss from 310 to 530 °C with an endothermal peak at 355 °C from DSC-TGA confirmed the presence of cellulose. Spherical cellulose nanocrystals (SCNCs) were produced using sulphuric acid hydrolysis with a size range of 50–190 nm. HPLC analysis of SCNCs confirmed the presence of only glucose and a minor fraction of cellobiose, indicating the high purity of the extracted cellulose.

Graphic Abstract



Keywords Baby diaper waste · Cellulose · Crystallinity · Characterisation · Extraction · Nanocellulose

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Statement of Novelty

The novelty presented in this work is the utilisation of used baby diaper waste to extract the cellulose in an ecofriendly approach. The demonstrated green approach could tackle the significant accumulation of diaper waste in municipal solid waste management. The present study also contributes for the synthesis of nano cellulose from the recycled cellulose which can be used for various applications. This is the first report on the untouched municipal solid waste, baby diaper waste for extracting the valuable polymer.

Introduction

The diaper is a type of comfort wear for babies to adsorb and retain their urine and faeces for a specified period. Diapers are broadly classified into disposable and reusable diapers based on their number of usage times. Disposable baby diaper comprises of 36.6% cellulose, 30.7% sodium polyacrylate, 16% polypropylene, 6.2% low-density polyethene and 10.5% elastic and adhesive tapes [33]. Disposable diapers are used for a single time and later discarded without recycling [26]. Disposable diaper wastes constitute 2–7% of total municipal solid waste that causes significant environment pollution [2]. In India, alone diapers and sanitary napkin waste comprises of 10% of municipal solid waste [16]. Globally, 3.5 million tonnes of diaper waste is discarded as solid waste that is either burnt or end up in landfills [15]. Major environmental issues of diaper waste include air pollution, global warming, fire outbreak, landfills, emission of foul odour, disease transmission from diapers with faeces, accumulation of landfills, leachate infiltration to groundwater, etc. [5, 14, 20]). Burning of diaper waste along with other solid waste releases toxic gases such as NO_2, SO_2, HCl, dioxins, CO_2, and ash into the atmosphere [5, 21]. Due to the presence of synthetic polymers, disposable diaper takes a maximum of 500 years to decompose [22] and contributes to the landfills. So, the baby diaper wastes recycle into cellulose and nanocellulose could pave a new path for tackling the massive generation of disposable diaper waste and produces a valuable biopolymer cellulose and nano crystalline cellulose.

Cellulose is the most abundantly available polymer on earth and used in food, pharmaceutical, fermentation and biofuel industries [36, 41]. Nanocellulose is cellulose with a size range of 20 × 100–200 nm and used in the manufacture of optical devices such as LCD TVs. Other applications of nanocellulose in anti-counterfeit technologies, particle tracking, production of greenhouse plastics, adsorbent, foams, filler for composites, and so forth also gaining interest in recent times [9, 36].

Extraction, characterisation and applications of cellulose and nanocellulose from the various lignocellulosic biomass have been reported [4, 10, 12, 13, 27, 36, 38]. But the systemic study for the extraction of cellulose and nanocellulose from the used baby diaper waste has not reported so far. But, the used diapers have been previously studied for the production of compost along with organic waste [6], methane using activated sludge [35], and hydrogen [7, 37]. Extraction of valuable biomaterials from waste is not only beneficial for the environment but also contributes to the circular economy. So the current work reports the recycling of 'diaper wastes' into cellulose and nanocellulose. The extracted cellulose can also be used again in the preparation of diaper apart from other applications.

The chlorine-based approach for cellulose isolation involves the use of acidified sodium chlorite. Despite achieving a good yield of cellulose, chlorite is highly toxic to humans as well as to the environment. When accidentally ingested in humans and animals, sodium chlorite causes methemoglobinemia, hemolysis, kidney injury and glutathione depletion [30]. When penetrates the soil, causes groundwater contamination. Hence an alternative and comparatively less toxic and environment-friendly chemicals for the cellulose isolation from the waste biomass are in high demand [29, 31, 34]. So, in the present study, a greener approach using a mixture of acetic and nitric acid was employed for the extraction of cellulose. The obtained cellulose and synthesised nanocellulose were analysed by FE-SEM, XRD, FTIR, DSC-TGA, TEM and HPLC.

Materials and Methods

Material and Chemicals

The samples of disposable urinated baby diapers of one single brand and size were collected from a local apartment, Thanjavur town, Tamil Nadu, India. Nitric acid (HNO_3, A.R 69%), acetic acid (CH_3COOH, Hi L.R 99.5%) and Sulphuric acid (H_2SO_4, Hi L.R 98%) were procured from Himedia, Mumbai, India.

Extraction of Cellulose

The urinated diaper was washed with tap water followed by autoclaving at 121 °C for 103.42 kPa for 20 min. The sterilised diaper was dried further in a hot air oven at 60 °C overnight. The swollen Super Absorbent Polymer (SAP) was removed manually, and the remaining diaper was size reduced in the range of 1 cm × 1 cm using a blender. From diaper waste, the cellulose was extracted as described by

[31]. Briefly, 3 g of the milled diaper was added to 60 mL of acetic acid (80%)–nitric acid (70%) mixture (1:20 w/v), with the ratio of acetic to nitric acid being 10:1 (v/v). The acid suspension was kept in a hot air oven at 120 °C for 15 min. Later the suspension was filtered using vacuum filtration with a 0.22 µm pore size filter membrane, and the filter cake, cellulose was collected. The obtained cellulose was repeatedly washed with distilled water, followed by ethanol to remove the excess acids. The washing step was repeated until the colour of cellulose became pure white.

Synthesis of Nanocellulose

Spherical cellulose nanocrystals (SCNCs) were produced by hydrolysing the isolated cellulose using H_2SO_4 [36]. After acid hydrolysis, the excess acid present in the cellulose was removed by dialysis, with a membrane cut off of 14,000 Da using double distilled water. Dialysis was followed by ultrasonication to obtain a stable mixture of nanocrystals. Detailed scheme of extraction of cellulose and synthesis of SCNCs was given in Fig. 1.

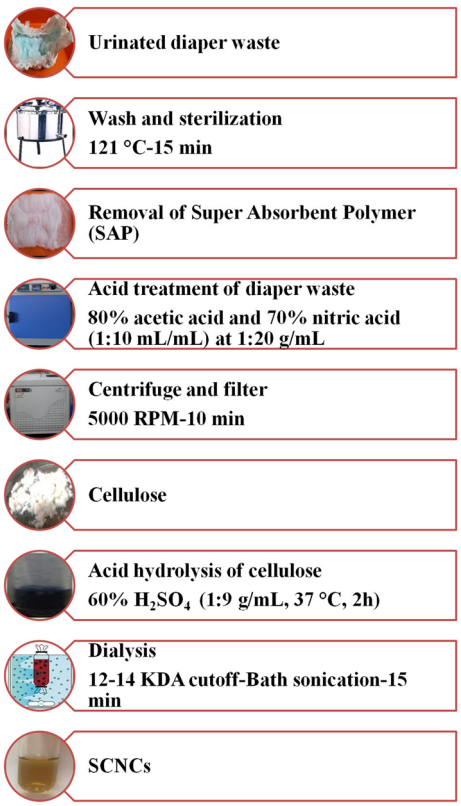

Fig. 1 The scheme for the extraction of cellulose from the diaper waste

Characterisation

Field Emission Scanning Electron Microscopy of the extracted cellulose from the diaper waste was imaged to analyse the surface morphology. A JEOL Field Emission Scanning Electron microscope (JSM 6701 F) was operated at a voltage from 5–10 kV. For analysis, the extracted cellulose was dried in a desiccator for 2 days and later mounted on a gold stub and observed in the microscope.

TEM analysis was performed to determine the morphology and size of the nanocellulose suspension. The sample was spread in a copper mesh and focussed using a JSM JEOL (2100 F) microscope. Digital micrograph software was used to process the images.

For zeta potential 291.6 Kcps count rate and 100 runs were performed at 25 °C using Malvern analytical zeta sizer (Malvern instruments). Regarding zeta size, the count rate was 271 Kcps and duration of 60 s was maintained at 25 °C.

The ^{13}C NMR (CP/MAS) spectrum of cellulose was analysed using a Bruker Avance HD 500 MHz spectrometer with D_2O at room temperature. The number of scans taken was 1024.

FTIR analysis was performed in a Perkin Elmer instrument (USA). The pellet sample was prepared with KBr, and the spectrum was analysed in the transmittance mode in the range of 400 cm^{-1} and 4000 cm^{-1} with a 1 cm^{-1} resolution.

Bruker D8 advance equipment was used to record the XRD pattern. The scanning range was 2θ angle from 5°- 90° with 0.2 s scanning time per step. A Cu kα X-ray source with a wavelength of 0.1540 nm was used. The following equations were used to determine the crystallinity index, crystal size, and d-spacing of the isolated cellulose.

The crystallinity index of the cellulose crystal was calculated as per Eq. 1 [18]

$$\text{C.I}\,(\%) = 100 \times \frac{A_{crystalline}}{A_{crystalline} + A_{amorphous}} \tag{1}$$

where $A_{crystalline}$ is the area of the crystalline peak, and $A_{amorphous}$ is the area of the amorphous peak.

Scherrer's equation can be used to determine the cellulose crystal thickness.

$$T = \frac{k\lambda}{\beta \cos\,\theta} \tag{2}$$

'λ' is the wavelength measuring 0.1540 nm, 'k' a correction factor of 0.91, θ is the angle of diffraction, β is full width at half maximum.

Bragg's law can be used to determine the d-spacing.

$$d = \frac{n\lambda}{2 \sin\,\theta} \tag{3}$$

Using TGA, the degradation pattern of the extracted cellulose for temperature was analysed in an SDT Q600 instrument. 5 mg of the sample was heated to 500 °C at 20 °C/min. Nitrogen was used as carrier gas with a flow rate of 100 mL/min.

The thermal behaviour of the extracted cellulose was also studied using a NETZSCH DSC 214 Polyma Differential Scanning Calorimeter instrument. 5 mg of the sample was heated to 400 °C at 25 °C/min. Nitrogen was used as carrier gas with a flow rate of 40 mL/min.

The presence of glucose in SCNCs was determined using HPLC (Waters) analysis. The spherisorb NH_2 column (4.6 × 250 mm) connected to a refractive index detector was used with acetonitrile (80%) as mobile phase at a flow rate of 1 mL/min of isocratic elution for the analysis.

Results and Discussion

Isolation of Cellulose

A chlorine-free method was used for the extraction of the cellulose from the diaper waste in the present study. A cellulose yield of 25.13 ± 1.02% w/w was obtained from diaper waste in the current work. Approximately 36% w/w cellulose is present in the diaper [33]. So, 11% cellulose which was unrecovered from the diaper waste, would have been partially hydrolysed in the acid mixture during the extraction process. The filtrate obtained after the extraction of cellulose mainly comprises of hemicellulose and polyethylene. Hemicellulose is a natural component present along with cellulose fibre [28]. The removal of hemicellulose from the diaper waste is essential as it contributes to the formation of crystalline cellulose. The acidic filtrate can be neutralised quickly and discarded, suggesting the simplicity and environmental friendliness of the method.

Morphology, Surface Charge and Size Characterisation

FE-SEM analysis of the extracted cellulose revealed long and twisted fibres which are typical for the cellulose [11]. The rough surface on the fibres denotes the extraction of hemicellulose and polyethylene. The flat shaped cellulose provides high surface area for the synthesis of various composites (Fig. 2a, b).

Nanocellulose was synthesised from the cellulose using sulphuric acid hydrolysis. Acid hydrolysis results in the destruction of amorphous regions in cellulose and exposes only the crystalline parts whose dimensions are in the nanoscale [13]. Nanocellulose generally exhibits needle shape with a diameter in the range of 10–20 nm [13, 31]. In the present work, spherical cellulose nanocrystals (SCNCs) as observed from the TEM analysis (Fig. 2c, d) with an average diameter of 50–190 nm and zeta potential of -5.84 mv were obtained. Nanocellulose generally exhibits needle-like morphology [31]. However, spherical shaped nanocellulose has also been previously reported [25, 36]. Spherical shaped nanocellulose is produced due to the degradation of amorphous cellulose. The SCNCs tend to accumulate as a result of water evaporation and thereby have a large size, varies from 20 to 300 nm [32]. The repulsion between identically charged particles in the suspension can be determined using zeta potential, which indicates the stability of a colloidal suspension [27]. A slightly low value of zeta potential in the current work suggests the presence of bigger agglomerated particles. SCNCs would either be present as a single particle or as agglomerated particles, thereby exhibiting a wide size range rather than uniform particle size [36]. Hence a difference in size range was observed in SCNCs analysed using TEM and zeta size.

CP/MAS ^{13}C NMR Spectroscopy

The CP/MAS ^{13}C NMR spectrum of the extracted cellulose was coherent with the previous reports (Fig. 3a) [36]. The cellulose resonances at 105.0 ppm represent carbon C1, 83.3, and 88.9 ppm for carbon C4, 62.6 ppm, and around 65.4 ppm indicate carbon C6, 72.2, and 74.9 ppm for carbons C2–C5. The signals at 88.9 and 65.4 ppm denote crystalline cellulose and the peaks at 83.3 ppm and 62.6 ppm attribute to disordered cellulose. The absence of peaks at 101, 80, 74, and 63 ppm indicates the absence of hemicellulose [23].

Functional Group Analysis by FTIR

The absorption bands of functional groups denoting cellulose were found to be coherent with the literature. The transmittance band at 3367 cm^{-1} and 2901 cm^{-1} belong to the stretching vibrations of –OH and –CH groups, respectively [27]. The –OH group of absorbed moisture in the extracted cellulose was observed at 1643 cm^{-1} [29]. The CH_2 bending of cellulose occurs at 1430 cm^{-1} [36]. The transmittance signal at 1373 cm^{-1} indicates O–H bending vibrations [29]. The C–O–C pyranose skeletal vibration occurs at 1164 cm^{-1}. The transmittance band at 1059 cm^{-1} is attributed to the antisymmetric bridge stretching vibration of C–O group [19]. The transmittance signal at 897 cm^{-1} is due to the glycosidic bonds of sugar molecules [13]. The transmittance peak at 1725 cm^{-1} indicates the minor presence of acetyl group of amorphous region of cellulose [24]. The bands at 1430 cm^{-1}, 1164 cm^{-1}, 1373 cm^{-1}, 1059 cm^{-1}, and 897 cm^{-1} confirms the presence of cellulose (Fig. 3b) and was consistent with the literature.

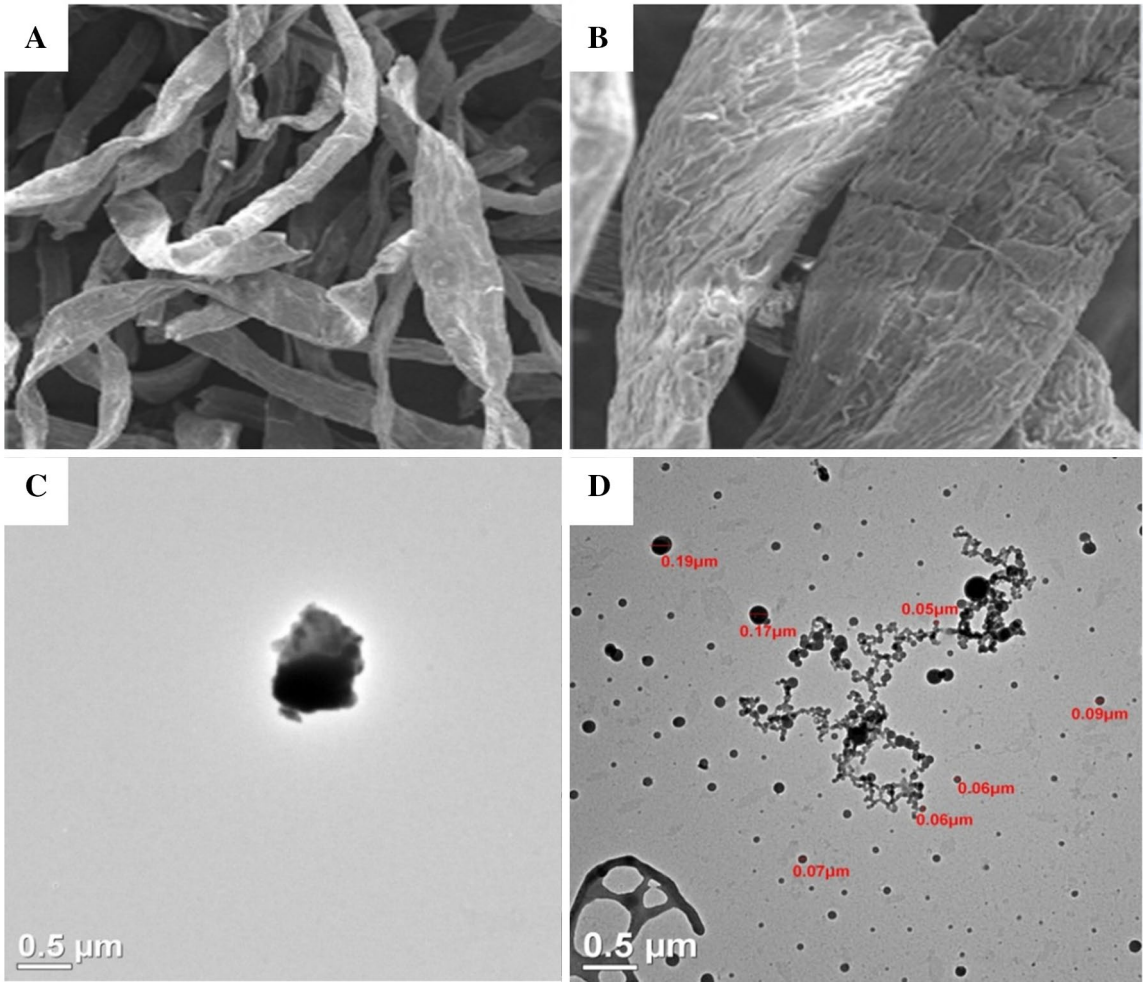

Fig. 2 SEM micrographs of cellulose **a** ×500 and **b** ×3000; TEM micrographs **c** single cellulose nanocrystal and **d** SCNCs in the range of 50–190 nm

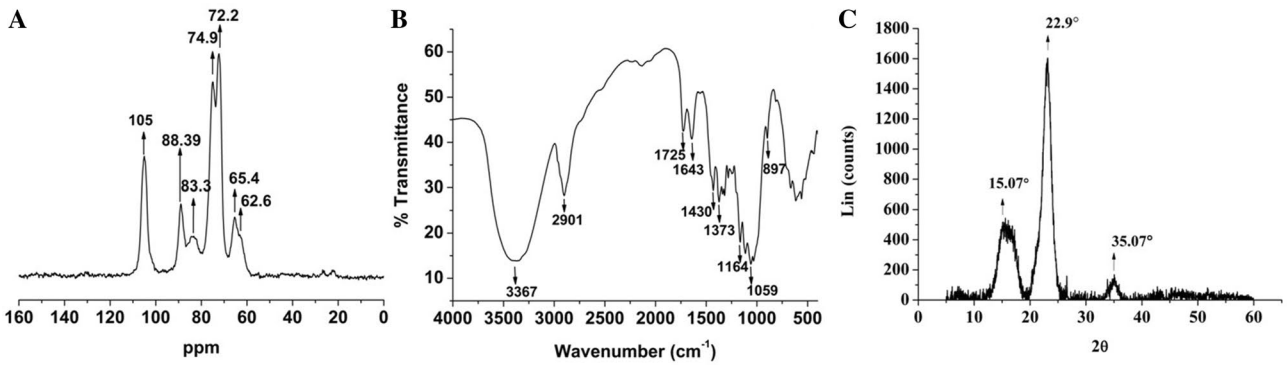

Fig. 3 Characterisation of the isolated cellulose from the diaper waste **an** NMR, **b** FTIR, **c** XRD

Crystallinity Study by XRD

The X-ray diffraction pattern of the extracted cellulose is shown in Fig. 3c. The cellulose patterns were observed at 15.07°, 22.9°, and at 35.07°, which corresponds to the lattice planes 110, 200, 004 of cellulose [3]. No doublet peak at 22.9° indicates that the extracted cellulose belongs to type I. From Eq. 1, the crystallinity of the cellulose in the present

study was determined to be 65.1%. The percent crystallinity index of the cellulose reported from rice husk whiskers was 67% [31], from wood 71%, potato tuber 66%, rice straw 68% [1] and sisal fibres 75% [24]. From Eqs. 2 and 3, the crystal size and interplanar distance of cellulose were found to be 4.24 nm and 0.19 nm.

Thermal Analysis by TGA and DSC

The TGA and DSC curves of the isolated cellulose are shown in Fig. 4a, b. The thermal degradation of the extracted cellulose was recorded in three phases. The first phase occurred during 29–137 °C and resulted in 7% weight loss indicating the volatization of moisture. The second phase of weight loss occurs at 300–370 °C. The characteristic depolymerisation of cellulose occurs during this period [17]. Maximum weight loss at this stage occurred at 368 °C. In the second phase alone, 67% weight loss was recorded. The third phase of weight loss occurred after 370 °C, which indicates the thermal decomposition of carbon residues. Degradation of cellulose stopped at 538 °C and the residue remaining are carbonaceous materials. The weight of the cellulose remaining after the third stage was only 16.2% indicating an 84% overall weight loss from the first to the third stage. Maximum weight loss for cellulose occurred in the second phase of degradation. The TGA pattern of the extracted cellulose was coherent with the literature [17]. The energy consumption property of the extracted cellulose was studied using DSC. The endothermal peak at 355 °C confirms the presence of cellulose [39]. Thus TGA and DSC peaks revealed for cellulose.

HPLC Analysis

The SCNCs displayed one large peak corresponding to glucose at 10th min and one smaller peak corresponding to cellobiose at 8.5th min (Fig. 4c). The results revealed that prepared SCNC is pure and is devoid of hemicellulose. Literature reports on HPLC analysis of nanocellulose are limited. Previous studies on HPLC analysis of nanocellulose has shown only the presence of glucose [10, 36]. The presence of cellobiose in the present work indicated the minor fraction of unhydrolysed cellulose in the SCNCs. The amount of acid required for the preparation of nanocellulose is around

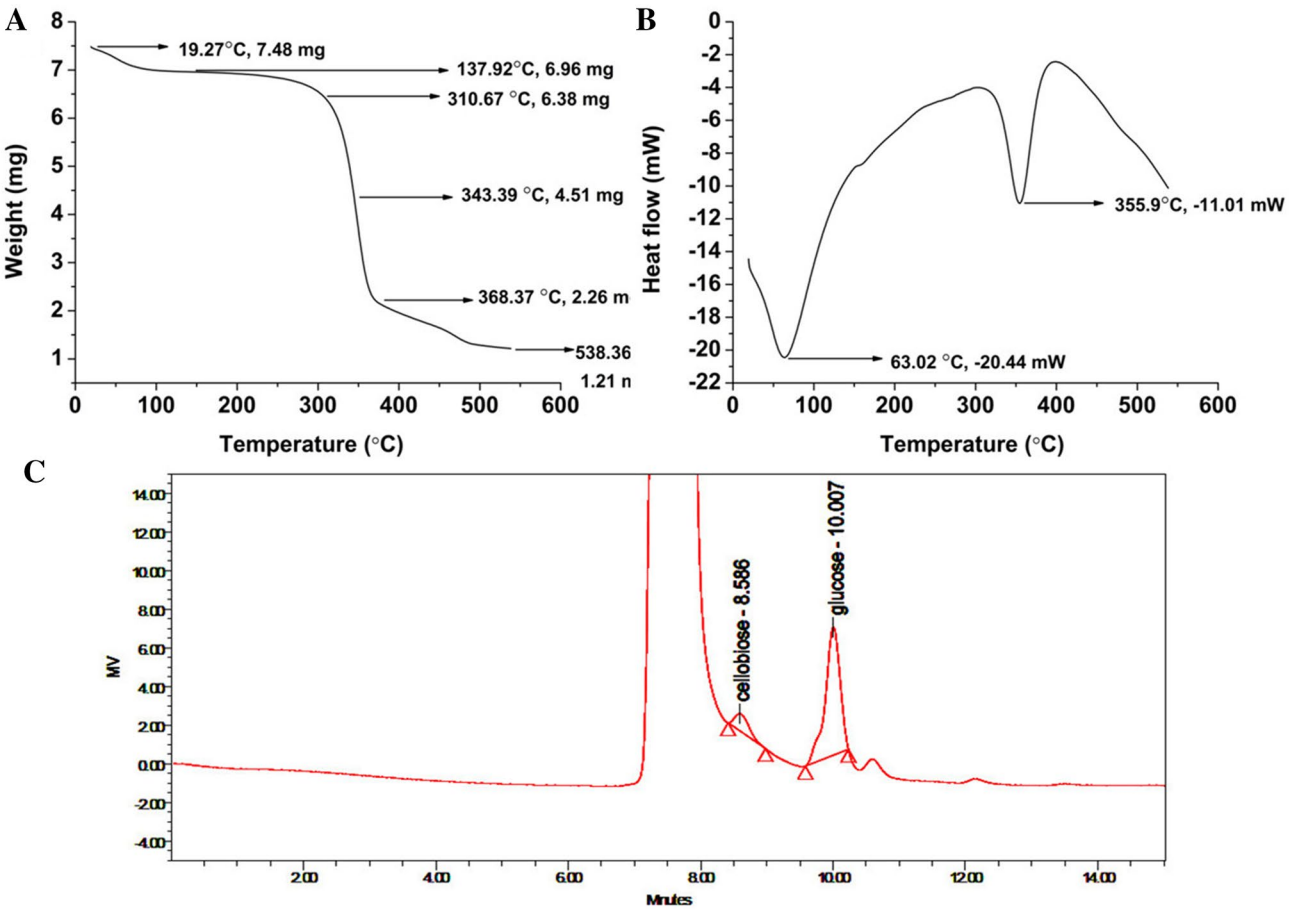

Fig. 4 Thermal analysis of the extracted cellulose from diaper waste **a** TGA, **b** DSC, **c** HPLC chromatogram of SCNs

60–64% [8, 25, 29, 40]. Despite using 60% H_2SO_4 in the present study, a minor fraction of unhydrolysed cellulose was present in the SCNCs, which could be acid-resistant cellulose.

Conclusions

The route for the synthesis of valuable materials from waste source is vital in the present scenario to combat environmental pollution. In this context, urinated disposable diaper, a waste material, was explored for the first time to produce the valuable polymer, cellulose. Cellulose fraction was isolated from the diaper waste using a green method. The use of less harmful acids, such as acetic acid and nitric acid, does not harm the environment as they can be easily neutralised. The amount of cellulose extracted from diaper waste in the present study was approximately 25%. FE-SEM, FTIR, XRD, TGA–DSC analysis of cellulose and TEM analysis of SCNCs were coherent with the literature. The current results indicate the significance of recycling of the used baby diaper for the production of cellulose and nanocellulose. The proposed green approach could tackle the significant accumulation of diaper waste in municipal solid waste management.

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Compliance with Ethical Standards

Conflict of interest All authors declare that they have no potential conflict of interest.

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