Chemical Papers

Surface modification and characterization of nanocellulose derived from the leaves of Borassus flabellifer

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Surface modification and characterization of nanocellulose derived from the leaves of *Borassus flabellifer*

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Abstract

Nanocellulose (NC) is revolutionizing the world of biopolymers. This research explores the potential of NC as an additive for polymer composites, focusing on enhancing their structural and physio-chemical properties. The study involves the extraction of NC from *Borassus flabellifer* leaves and surface modification using lactic acid (LA). The composition of the leaves was estimated to be 62.2% cellulose, 15.8% hemicellulose, and 12.2% lignin by weight %. NC was extracted by subjecting pre-processed leaves to alkali treatment, resulting in an average particle size of 317 nm. NC and surface-modified NC (SMNC) were characterized using various bioanalytical techniques, including SEM, FTIR, and XRD, to evaluate the effectiveness of the surface modification. The water absorption test was performed to assess the surface modification of NC. The results confirmed the successful surface modification of NC using LA, as evidenced by the presence of LA functional groups on the NC after modification. XRD analysis confirmed the crystalline nature of NC and SMNC, with a higher crystalline index of 48.27% for SMNC compared to 42.78% for NC. The water absorption activity of SMNC was also measured and found to be lower than that of NC, suggesting that SMNC may be a promising additive for polymer composites. Overall, this study demonstrates the potential of SMNC for various applications in materials science.

Keywords *Borassus Flabellifer*. Nanocellulose. Surface modification. Lactic Acid

Introduction

Natural fibers are a renewable and sustainable source of biodegradable materials that offer several advantages over synthetic fibers. They are lightweight, low cost and widely available, making them an attractive option for use as reinforcements in polymeric matrices. By extracting natural fibers from various plant parts, such as the bark, stem, root, fruits, leaves and flowers, new bio-based composites can be created with enhanced properties. *Borassus fabellifer* is among the sources of natural fiber that can be used as biodegradable materials [1]. *Borassus flabellifer*, a member of the Arecaceae family, is native to Africa but is primarily grown in Indo-Malaysia and Australia. It is usually referred to as the Palmyra palm. *Borassus flabellifer* leaves are used to make umbrellas, mats, hats, fans, and writing material; nevertheless, different parts of the plant have diverse scientific qualities that may be utilised in different dimensions of research. For example, cellulose from the leaves has many applications in the food packaging industry [2]. Cellulose, a polysaccharide composed of linear D-glucose chains linked together by β-1,4-glycosidic bonds, is one of the significant components of the leaves of *Borassus flabellifer*. Natural fibers, which include cellulose, hemicellulose, and lignin, are commonly utilized by biopolymers to improve their physicochemical properties [3]. Among these, cellulose is the most abundant and primarily produced by plants. Natural fibres can be purified for biopolymer applications through alkali treatment, which selectively removes impurities such as hemicellulose and lignin, leaving behind pure cellulose. This treatment effectively reduces the presence of hydrophilic components on the surface of the fiber, increasing cellulose content [4].

One of the unique substances derived from cellulose is NC, which is obtained through various extraction methods. Due to its greater surface area, aspect ratio, and Young's modulus, NC has an advantage over ordinary cellulose fibers [5]. The properties of NC are determined by the source, the isolation method, and subsequent surface modifications [6]. NC is reinforced

into various biopolymers to improve its physio-chemical properties [7,8]. The properties such as biocompatibility, biodegradability, and adaptable surface chemistry make it suitable for biopolymer reinforcement [9]. The NC added as an additive is thought to improve the strength and stiffness of the biocomposite formed, thereby improving the probability of its industrial use [10].

Moreover, being biodegradable, NC is considered safe for use. However, unmodified NC has certain disadvantages, making it less suitable for some applications. One of the major drawbacks of unmodified NC is its hydrophilic nature. Hence, it has a strong tendency to absorb moisture. This high water absorption rate can reduce the mechanical properties of materials made from NC, making it less suitable for applications where moisture resistance is crucial, such as in packaging materials. The high water absorption rate can also lead to swelling, which can cause dimensional changes in the material, affecting its overall stability and durability. Furthermore, due to its hydrophilic nature, NC has poor compatibility with hydrophobic polymers, such as polylactic acid (PLA) [11]. This lack of compatibility can result in phase separation and reduced interfacial adhesion between NC and the hydrophobic polymer matrix. This can lead to decreased mechanical strength and stiffness of the resulting composite material. Another disadvantage is its tendency to form aggregates due to its high surface area and reactivity, which can lead to difficulties in processing and handling [12]. These aggregates can also cause non-uniform dispersion of NC in the polymer matrix, which can further affect the mechanical and physical properties of the resulting material. Surface modifications are, therefore, performed to overcome these disadvantages. Specifically to reduce the hydrophilicity of NC [13]. These modifications can improve its compatibility with polymer, improving the resulting nanocomposites' mechanical, thermal, and barrier properties. Introducing ester groups can also improve the dispersion of NC in polymer, reducing the aggregation and improving the interfacial adhesion between the two materials. This can lead

to more sustainable and eco-friendly materials with improved properties, making them suitable for various applications, such as packaging, biomedical devices, and drug delivery systems.

Although numerous researchers have documented the production of NC from *Borassus flabellifer* as an additive. A very limited study has been conducted on surface modification of NC extracted from the leaves of *Borassus flabellifer* using LA and its use as an additive for polymer composite. This study aims to synthesise the NC from the leaves of *Borassus flabellifer* and surface modify it with LA [14] so that it can be used as an additive for enhancing the mechanical, physio-chemical, and gas barrier properties of biopolymer and its widespread applications.

Experimental

Materials

HCl, NaOH, H_2O_2, LA and $SnCl_2$ were used as a commercial-grade reagent from Sisco Research Laboratories Pvt. Ltd, India. *Borassus flabellifer* leaves were gathered from Potheri, Chennai, India. NC was extracted from these leaves, and it was subsequently modified to SMNC.

Proximate composition of *Borassus flabellifer* leaves

Cellulose, hemicellulose, and lignin content were estimated for *Borassus flabellifer* leaves. The chemical compositions were estimated using gravimetric analysis. Equation (1) was used to calculate the chemical composition of cellulose [15], hemicellulose [16] and lignin [1].

$$Wt\ (\%) = \frac{(H1-H2)}{H1} \times 100 \quad \text{.....(1)}$$

Where,

H1 is the weight of the untreated leave, and H2 is the weight of the treated leaves.

Pre-processing and pre-treatment of *Borassus flabellifer* leaves

The leaves samples were collected and processed further. The pre-processing of leaves was performed by mechanical operation technique known as ball milling. The obtained powder was alkali treated with 4% NaOH (1M) solution at a ratio of 1:10 and incubated at 80 ℃ for 3 h, followed by filtration. The alkali-treated biomass was bleached with 3% H_2O_2 and 4% NaOH (1M). The sample was again incubated at 80 ℃ for 3 h, followed by filtration and collection of the biomass. The biomass was ultrasonicated for 30 min at 20 kHz with a power input of 750 W, with an amplitude of 30% and pulses of 10 seconds on and off [17]. Subsequently, the samples were kept, and NC extraction was carried out.

Synthesis of NC

The ultrasonicated sample was centrifuged for 12 min at 10,000 rpm, and the supernatant was collected. The collected supernatant was lyophilized for 4 h for converting into powder form.

Surface modification of NC with LA

In the presence of the $SnCl_2$ (100 µg), NC aqueous solution was blended with LA at a ratio of 15:1. The sample was homogenized at 5000 rpm for 10 min, followed by ultrasonication for 1 h at 20% amplitude with a pulse of 10 sec on and off. The processed NC was kept in an 80°C water bath for 10 h. The sample was then filtered and air-dried [18].

Characterization studies of NC and SMNC

The extracted NC was characterized with a zeta sizer to confirm the nanoform. To confirm the particle size, the extracted NC was examined at 516 nm, 517 nm, and 518 nm

wavelengths [19]. The untreated leaf, untreated leaf with mechanical operation, alkali-treated leaf, NC and SMNC were then analyzed using SEM at 40,000 X magnification to determine surface morphology [20]. Following that, NC and SMNC were analyzed by FTIR and XRD. FTIR findings revealed the existence of functional groups. KBr pellets were used to prepare the samples with 10 mm in diameter and 1 mm in thickness for FTIR analysis. 5 mg of NC and SMNC were analysed by Agilent Technologies, Cary 600 Series / a Perkin-Elmer infrared spectrophotometer between the frequency range of 400–4000 cm^{-1}. The crystalline/amorphous nature of the particles and the crystallinity index were determined by performing XRD analysis at room temperature with a wavelength of 1.54060 Å with a Cu-Kα source and a generator at 40 kV 15 mA [21]. The crystallinity index (CrI) was calculated using the diffraction intensities of the crystalline structure and the amorphous fraction. Eqn. 2 is used to calculate the crystallinity index,

$$\mathrm{CrI\%} = \frac{[\,(I_{002} - I_{am})]}{I_{002}} * 100 \quad \ldots\ldots\ldots(2)$$

Where,

I_{002} is the maximum intensity of the diffraction peak, and I_{am} is the intensity of the amorphous diffraction peak [22].

Water absorption test of NC and SMNC

The water absorption test was conducted using the gravimetric method. 1 g of NC and SMNC was taken, and their dry weight was recorded. Both samples were dipped in 50 mL of water. Furthermore, the weight change was recorded every 5 min until it obtained a constant weight. Water absorption was determined by measuring the increase in mass as a percentage of dry mass, and the moisture content value was calculated [23].

Results and Discussions

Proximate composition of *Borassus flabellifer* leaves

The chemical composition of cellulose, hemicellulose and lignin in untreated Borassus flabellifer leaves was evaluated using the gravimetric approach. Estimates showed that the leaves contain 64.2% cellulose, 15.8 % hemicellulose, and 12.2% lignin. Similar findings were reported by Singh et al. for untreated *Borassus flabellifer* leaves, with 68.1% cellulose, 14.5% hemicellulose, and 11.5% lignin contents [1].

Zetasizer Analysis

The Zetasizer is a powerful analytical instrument used to measure the size of particles in solution. The instrument utilizes dynamic light scattering (DLS) to determine particle size. To validate the isolated NC's particle size, zeta analysis was performed. The extracted NC's particle size distribution is depicted in Fig. 1. The NC isolated from leaves of *Borassus flabellifer* has particles with a diameter of 317 nm. Phanthong et al. [24] demonstrated that the particle size of NC ranges from 100 nm to 1000 nm, and a similar type of result was obtained for NC isolated from the leaves of *Borassus flabellifer*.

SEM Analysis

Scanning Electron Microscopy (SEM) is a powerful analytical technique used to investigate samples' surface morphology and composition. SEM analysis was performed to analyze the surface morphology of the untreated leaf, untreated leaf with mechanical operation, alkali-treated leaf, extracted NC and SMNC. The images were recorded at 40,000 X (Fig. 2). The SEM image recorded that the untreated leaf has a flat and sleek surface, untreated leaf with mechanical operation has large particles, while damaged and clump types particles were ascertained for alkali-treated leaf. An analogous kind of picture was observed by Arasu et al.,

following a sleek surface for untreated leaves and a rough surface for treated leaves [25]. On the other hand, the SEM images depicted that NC has a smooth and shiny surface for small particles, whereas SMNC particles have a clump-like pattern with a rough surface. The rough surface with a clump formation indicates that LA has bound with NC. According to Wei et al., surface modification of NC affects the degree of smoothness of the NC, resulting in a rough surface with clump-like development. When NC was modified with LA, a similar configuration was detected. It suggests that the surface modification has caused changes at the nanoscale level, which may have implications for the material properties and potential applications of NC [18].

FTIR Analysis

Fourier Transform Infrared Spectroscopy (FTIR) is a technique used to identify and analyze the chemical composition of a sample. In FTIR analysis, a beam of infrared light is directed through the sample, and the energy absorbed by the sample is measured. The resulting spectrum provides information about the functional groups present in the sample. FTIR spectra were used to investigate the functional groups present in the NC, LA, and SMNC. The effect of esterification for NC and SMNC through LA is shown in Fig. 3. The FTIR spectra show a prominent peak for NC between 3000 and 3500 cm^{-1} and at 1637.53 cm^{-1}, confirming the presence of functional groups in NC. The peak between 3000 and 3500 cm^{-1} presents evidence for the OH stretching of the free OH groups on the cellulose, and the peak at 1637.53 cm^{-1} represents the C-O stretching of the NC. The peaks obtained for LA were similar to those observed by Devi et al. [26]. Followed by, FTIR analysis of SMNC was performed, which revealed that the surface modification occurred due to a shift in the intensity between the functional groups. The intensity of the OH group was seen to be reduced in SMNC compared to that of NC. The acidic group reacted with the alcohol group, resulting in the esterification

process. Moreover, a carbonyl group formation at 1720 cm^{-1} has been observed in the SMNC with LA, demonstrating that the surface modification was effective from a chemical standpoint.

XRD Analysis

X-ray diffraction (XRD) analysis is a powerful technique used to determine a crystalline material's atomic and molecular structure. XRD works by shining a beam of X-rays at a sample and then measuring the angles and intensities of the scattered X-rays. This data can be used to calculate the positions of the atoms in the sample and their arrangement within the crystal lattice. XRD analysis of NC and SMNC revealed the crystalline and amorphous nature of the particles. The XRD pattern revealed that SMNC had a better crystalline structure than NC (Fig. 4). A notable peak of about 21.75° and 21.83° was observed for both NC and SMNC, respectively, confirming the findings of the study by Arun et al. [17]. The notable sharp peak in SMNC revealed the crystalline nature of the particle. While no such prominent peak was observed for NC, except a small peak around 21.75°. Further, the crystallinity index (CrI) of both NC and SMNC was evaluated as 42.78 % and 48.27 %, respectively. This was confirmed by the results of Robles et al. [27], who observed the crystallinity index of SMNC to be 53 %.

Water absorption test

The water absorption test is a widely used method for measuring the porosity and permeability of materials. The test involves immersing a sample of the material in water for a specific period and then measuring the amount of water the material has absorbed. The water absorption test is used to assess the durability and resistance of a material to weathering, erosion, and other forms of deterioration. The water absorption of NC and SMNC was studied and represented in Fig. 5. In the first 5 min, rapid water uptake was observed for both samples. Afterwards, the water uptake was reduced in the next 20 mins, after which a constant weight was observed between 30 min to 50 min. The NC sample showed higher water absorption as

compared to the SMNC. The result showed a 30 - 32 % reduction in water absorption of SMNC in comparison to that of NC. The observed reduction of water absorption for SMNC was comparable with the findings of Sethi et al. [28], who observed a 33 – 35 % reduction in water absorption of LA-modified NC compared to NC. With this observation, it can be pointed out that the surface modification of NC was successful, as reduced water absorption is vital for packing material.

Conclusion

Natural fibers, including cellulose and its derivatives, have attracted considerable attention as eco-friendly alternatives to synthetic materials, particularly in packaging applications. As a renewable and abundant resource, cellulose-based materials offer several advantages, including biodegradability, low toxicity, and high strength. Furthermore, the development of NC has expanded the potential applications of cellulose-based materials by providing enhanced properties such as high surface area, aspect ratio, and mechanical strength. In this study, NC was synthesized via ultrasonication from pre-processed *Borassus flabellifer* leaves, and its average size was determined to be 317 nm using a zeta sizer. The synthesized NC was then surface-modified using lactic acid (LA) through esterification in the presence of SnCl2. The resulting modified NC (SMNC) was characterized using various analytical techniques such as SEM, FTIR, and XRD. The SEM analysis revealed the binding of LA with NC, while the FTIR spectra confirmed the successful surface functionalization of the synthesized NC through the presence of ester carbonyl peaks. The XRD analysis indicated the particle nature of NC and SMNC. Furthermore, the water absorption test revealed that NC had a higher water absorption capacity than SMNC, suggesting that the esterification of NC successfully reduced its water absorption, which is crucial for packaging materials. Overall, it highlights the importance of natural fibers, cellulose, and NC in developing sustainable materials for various applications, including packaging. Further research in this area has the

potential to lead to the development of eco-friendly materials that can reduce the environmental impact of packaging waste while maintaining the desired performance characteristics. The findings indicate that the surface modification of NC through esterification provides an ecologically friendly technique to improve its physio-chemical characteristics. SMNC can be blended with biopolymers such as polylactic acid (PLA) to create sustainable packaging materials for food and beverage items. This research contributes to developing eco-friendly packaging materials, potentially reducing the environmental impact of packaging waste.

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Ethical Approval

Not applicable

Consent to Participate

The authors have agreed to participate in the publication of the paper

Consent to Publish

All authors have agreed to publish the paper

Authors Contributions

Ankit Chakraborty: Formal analysis of data, validation and preparation of original draft; **Pradnya Ghalsasi**: Collection of resources like protocols, and other related research papers and reviewing original draft; **Radha P**: Conceptualization, supervision, writeup revision - review and editing.

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Not applicable

Competing Interests

The authors declare no conflict of interest.

Availability of data and materials

The data used to support the findings of this study are available from the corresponding author upon request

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Figure legends

Fig. 1. Zeta-sizer analysis of nanocellulose

Fig. 2. SEM analysis of (a) Untreated leaf, (b) Untreated leaf with a mechanical operation, (c) alkali-treated leaf, (d) nanocellulose, (e) Surface modified nanocellulose

Fig. 3. FT-IR analysis of (a) nanocellulose, (b) lactic acid, (c) surface modified nanocellulose

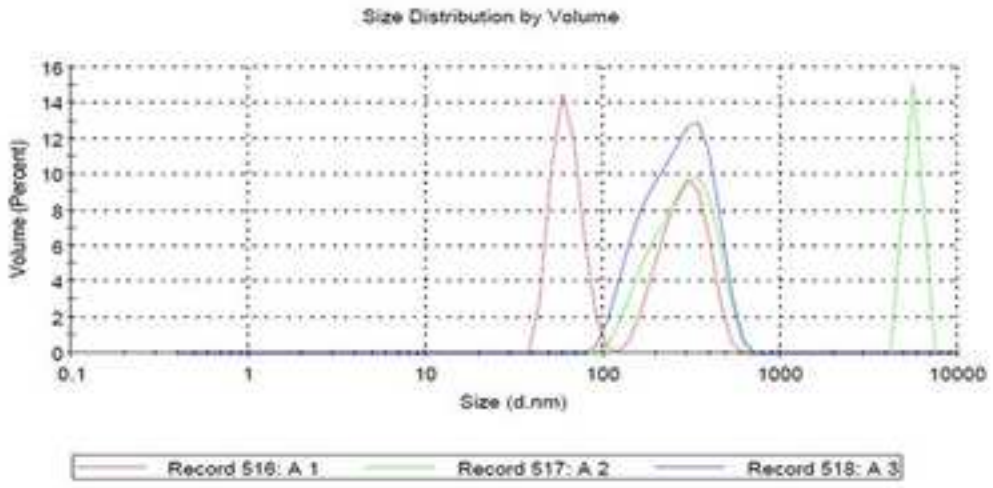
Fig. 4. XRD analysis of (a) nanocellulose (b) surface modified nanocellulose

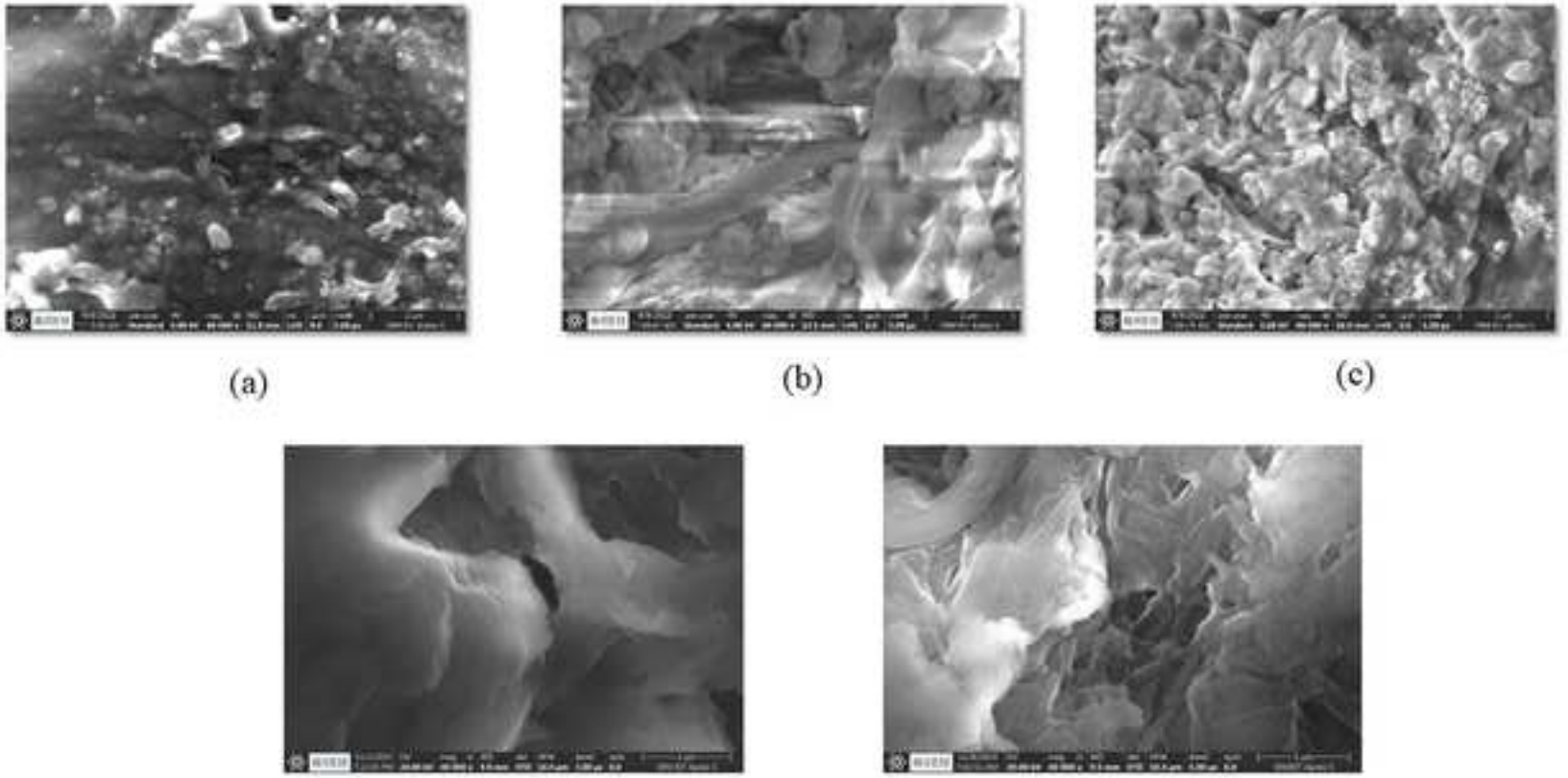
Fig. 5. Water absorption test of (a) nanocellulose (b) surface-modified nanocellulose

Figure

Results

		Size (d.nm):	% Volume:	St Dev (d.nm):
Z-Average (d.nm): 317.0	Peak 1:	292.5	51.9	83.11
PdI: 0.490	Peak 2:	62.88	48.1	12.29
Intercept: 0.920	Peak 3:	0.000	0.0	0.000
Result quality Good				





Figure

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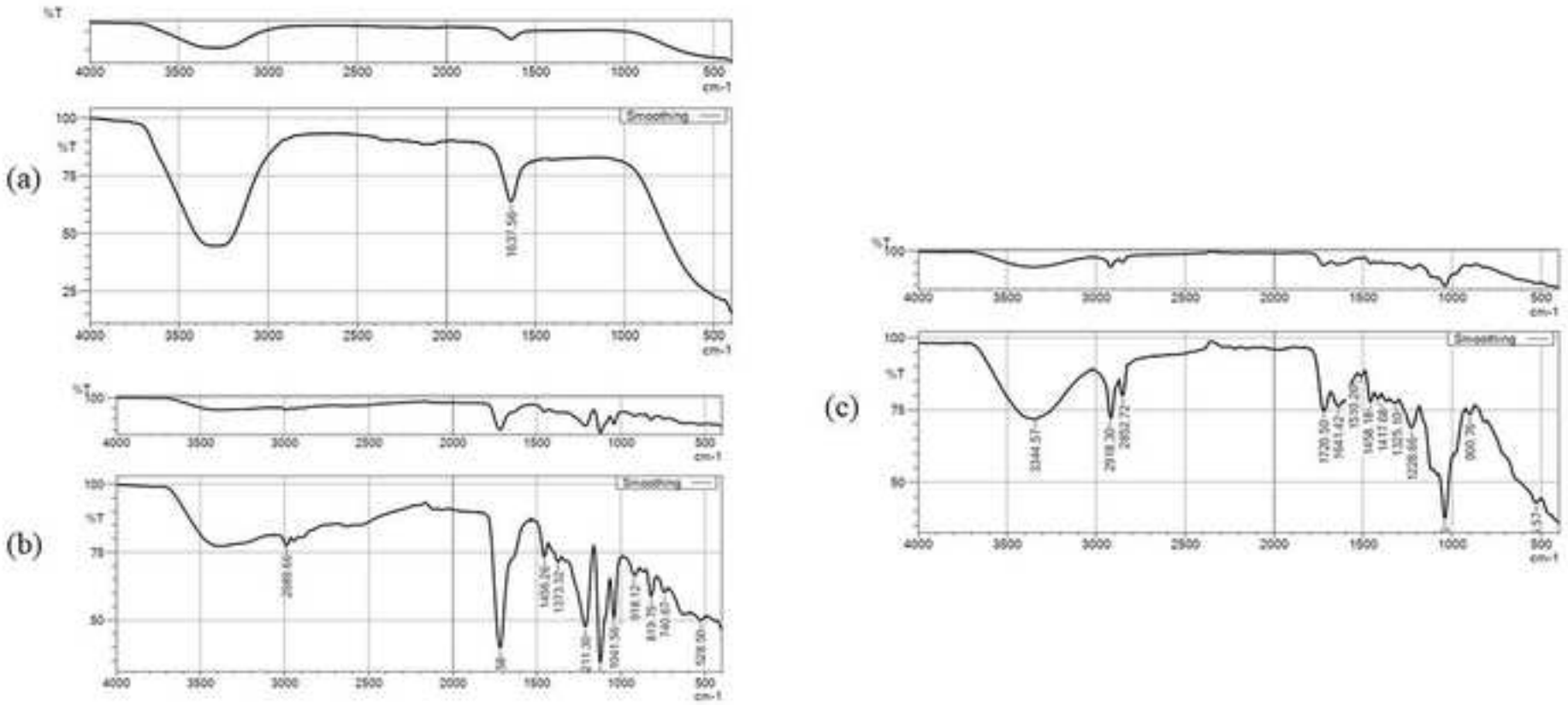

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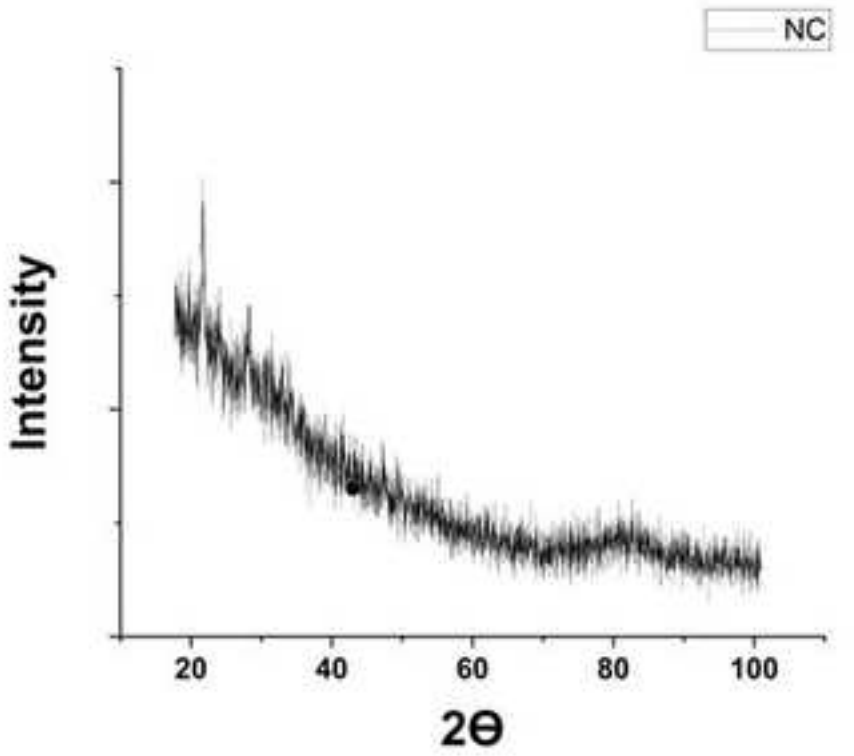
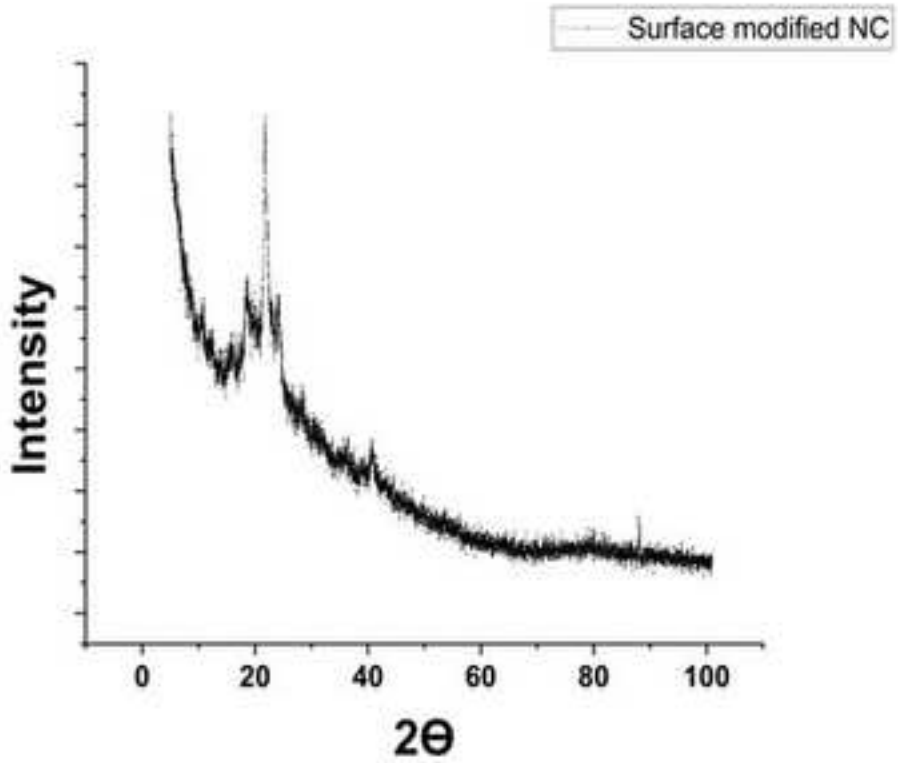


(a)



(b)

Figure

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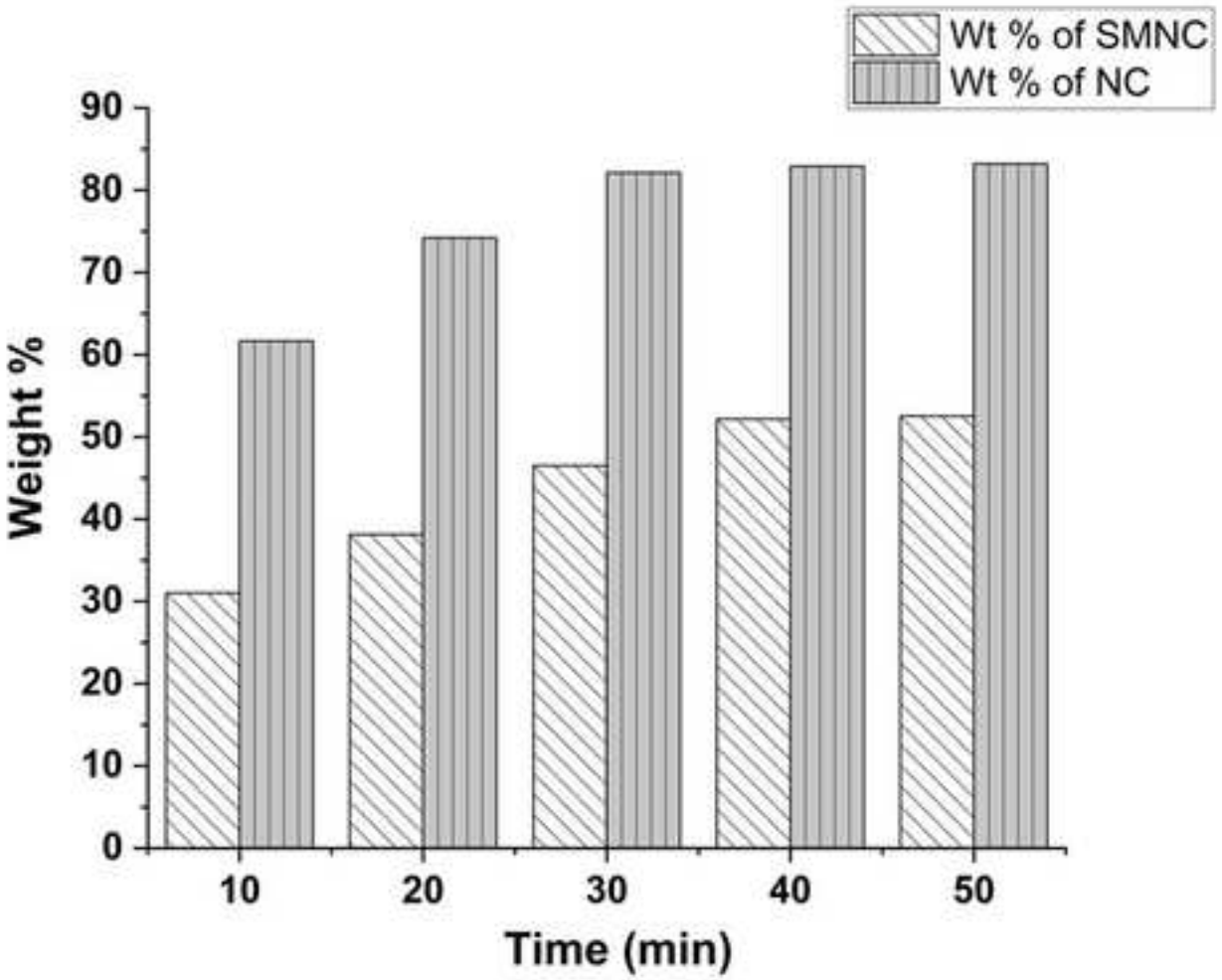

Graphical abstract