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Original Research Article

Preparation and Characterization of Cellulose Nanoparticles from Agricultural Wastes and Their Application in Polymer Composites

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Abstract

Background: The development of innovative eco-sustainable bio-nano-composites materials based on cellulose nanomaterials obtained from agricultural waste such as rapeseed plant straw and polymeric materials with improved properties. At first, cellulose was extracted from rapeseed plant straw and cellulosic and no cellulosic materials were estimated. It was found that the rapeseed plant straw contained about 43.48 wt.% cellulosic materials and 56.52 wt.% no cellulosic materials. It was also found that the rapeseed plant straw content about 34.84 wt.% α- cellulose. Then, nano-cellulose was prepared by acid hydrolysis of α -cellulose obtained from rapeseed plant straw. Poly vinyl acetate (PVAc) was prepared by soap free emulsion polymerization of vinyl monomer. Natural rubber (NR) collected as latex was oxidized by $KMnO_4$. Blends of NR and oxidized NR with PVAc of different compositions such as NR60/PVAc40, NR40/PVAc60, ONR60/PVAc40, ONR40/PVAc60 were prepared by mixing NR latex and PVAc emulsion and followed by drying in an oven at 60°C. Composites of NR60/PVAc40 and ONR60/PVAc40 reinforced nano-cellulose (NC) were also prepared in similar way. The blends and composites were then characterized by FTIR, SEM, and Tensile test (TS). FTIR analysis confirmed the oxidation of NR and shown the variation of the functional groups in the blends and composites compared with the FTIR spectrum of NR, ONR and PVAc. SEM study exhibited that the nano-cellulose were dispersed within NR/PVAc and ONR/PVAc blends matrices with a little agglomeration. The tensile test results showed that the strength in composites was increased due to the reinforcing effect of nano-cellulose (NC). ***Objectives:*** The aim of this study is to assess the Preparation and characterization of binary blends of NR and oxidized NR with PVAc. ***Methods:*** This is an observational study. The study used to be carried out in the admitted patient's Department of Applied Chemistry and Chemical Engineering, Islamic University, Kushtia-7003, Bangladesh. The duration of the period from Data was entered in MS Excel and Statistical analysis was done using SPSS trial version. ***Results:*** This study shows that the according to Constituent, Cellulose was 43.4822%, Alpha-Cellulose were 34.8427% and Cellulose were 8.6395%. And according to Source, Rice Straw were 32.15% %, Sugarcane were 41-43%, Rye Straw were 31.8-42.64%, Corn Stalks were 29.80% and Wheat Straw were 34-40%. ***Conclusion:*** Novel eco-friendly bio-nano-composites substances primarily based on renewable and sustainable assets namely natural rubber (NR) and nano-cellulose received from rapeseed plant straw as properly as poly vinyl acetate (PVAc) have been developed. In order to enhance the interfacial interactions amongst the composite factors such as NR, PVAc and NC, oxidation of the NR latex was once carried out by way of $KMnO_4$ before blending mixing with PVAc emulsion and NC suspension.

Keywords: Cellulose; Nanomaterials; Natural rubber (NR); Poly vinyl acetate (PVAc); Tensile test (TS).

INTRODUCTION

Each and every year Bangladesh, as an agricultural country, produces a large number of agricultural wastes and residues such as rice straw, rice husk, wheat straw, banana rachis, rapeseed straw etc. these agricultural residues and wastes are usually used to burn for heat, to prepare natural manure, and to feed domestic cattle by rural peoples [1]. These wastes are considered as lignocellulosic materials as cellulose and lignin are the major components of these wastes. Upgrading of such wastes through the development of innovative products such as cellulose nanomaterials and nanocomposites can be of high environmental, economic and social interest [2]. Besides, these wastes can be a sustainable resource in polymer composites and nanocomposites as sustainability and biodegradability of the polymeric materials become a great important issue because of limited reserve of petroleum and concerns related to the environmental pollution of non-degradable plastic waste [3]. Hence, proper utilization of these wastes through the development of innovative composite materials can protect the environment from pollution of them in one hand and on the other hand these wastes can be used as a sustainable and renewable resources in polymeric materials which can reduce the dependency of polymeric materials on petroleum [4].

Cellulose nanomaterial obtained from agricultural wastes were planned to be used for the development of novel bio-nanocomposites with natural rubber (NR) latex and poly (vinyl acetate) (PVAc) as matrices based on reactive compatibilization approaches [5]. Sustainable and biodegradable polymer blends and nanocomposites with improved mechanical properties were expected to be achieved. Moreover, it was also expected that production of value-added nanomaterials from agricultural waste and application of NR in new sector also find a new route for the economic development of Bangladesh [6].

However, the preparation of nanocomposites based on blends seems to be new in nanocomposites studies reported their mechanical properties and morphology of nanocomposites. This was why, before blending NR latex with PVAc emulsion and NC suspension, NR were planned to be oxidized to insert hydroxyl group on the NR chain [7]. A chemical interaction among composite components i.e. oxidized NR, PVAc and NC was expected. In this paper, preparation and characterization of NR and PVAc blend and their composite with NC have been reported.

METHODS

The rapeseed plant straws used as raw material were collected from a farmer of Pabna district. Natural rubber latex was collected from Cox's bazar (Ramu). Vinyl acetate monomer and other chemicals used in this paper as listed were purchased from Nafi-nano Science and some required chemicals were also provided from the department of Applied Chemistry and Chemical Engineering, Islamic University, Kushtia, Bangladesh.

Various efficient methods have been developed by researchers to isolate cellulose from lingo-cellulosic materials. Cellulose is isolated by the removal of other substances such as hemicelluloses and lignin from straws.

For this investigation the rapeseed plant straw was collected from Pabna district region. When rapeseed seed got matured then the seeds were collected from the field and the plant straw were rejected as agro waste. The straws were dried in air exposing sunlight. These straws were used for the extraction of cellulose.

RESULTS

Table 1: **Average compositions of Brassica napus (Rapeseed plant) plant straw**

Constituent	Weight (%)
Cellulose	43.4822
Alpha-Cellulose	34.8427
Cellulose	8.6395

Table 1 demonstrated and distribution of the Average compositions of Brassica napus (Rapeseed plant) plant straw. Here according to Constituent, Cellulose was 43.4822%, Alpha-Cellulose were 34.8427% and Cellulose were 8.6395%.

Table 2: Cellulose content in other plant straws

Source	Cellulose
Rice Straw	32.15% [Shawky et al.2011]
Sugarcane	41-43% [Mokhena et al.2018]
Rye Straw	31.8-42.64% [Kraszkiewicz et al.2015]
Corn Stalks	29.80% [Shawky et al.2011]
Wheat Straw	34-40% [Kapoor et al.2016]

Table 2 demonstrated and distribution of the Cellulose content in other plant straws. Here according to Source, Rice Straw were 32.15% %, Sugarcane were 41-43%, Rye Straw were 31.8-42.64%, Corn Stalks were 29.80% and Wheat Straw were 34-40%.

Table 3: FTIR spectrum analysis of NR

Band intensity wavenumber	Wave number cm-1	Absorption	Assignment
Strong	2957	C-H	Stretching vibration
Strong	2929	CH	Stretching vibration
Medium	1644	C=C	Stretching vibration
Strong	1457, 1364	C-H	CH_2 and CH_3 deformation
Strong	834	C=C	C=C bending

Table 3 demonstrated and distribution of the FTIR spectrum analysis of NR. When Absorption were C-H, Band intensity wavenumber were Strong, Wave number were 2957 and Assignment were Stretching vibration. When Absorption were CH, Band intensity wavenumber were Strong, Wave number were 2929 and Assignment were Stretching vibration. When Absorption were C=C, Band intensity wavenumber were Medium, Wave number were 1644 and Assignment were Stretching vibration.

Table 4: FTIR spectrum analysis of oxidized natural rubber

Band intensity wavenumber	Wavenumber cm-1	Absorption	Assignment
Strong	3200	O-H	Stretching vibration
Strong	2907	C-H	Stretching vibration
Medium	2343	O=C=O	Stretching vibration
Strong	1715	C=O	Stretching vibration
Strong	1616	C=C	Stretching vibration
Medium	1447	C-H	C-H bending
Medium	1390	O-H	O-H bending
Medium	1172	C-O	C-O stretching

Table 4 demonstrated and distribution of the FTIR spectrum analysis of oxidized natural rubber. When Absorption were O-H, Band intensity wavenumber were Strong, Wave number were 3200 and Assignment were Stretching vibration. When Absorption were O=C=O, Band intensity wavenumber were Medium, Wave number were 2343 and Assignment were Stretching vibration.

When Absorption were C=O, Band intensity wavenumber were Strong, Wave number were 1715 and Assignment were Stretching vibration. When Absorption were C-H, Band intensity wavenumber were Medium, Wave number were 1447 and Assignment were C-H bending.

Table 5: FTIR spectrum analysis of PVAc

Band intensity wavenumber	Wavenumber **cm-1**	**Absorption**	**Assignment**
Weak	2930	C-H	Stretching vibration
Weak	2343	O=C=O	Stretching vibration
Strong	1725	C=O	Stretching vibration
Medium	1365	C-H	Bending vibration
Strong	1226	C-O	Stretching vibration
Strong	1023	C-C	Stretching vibration
Medium	625	C-H	Rocking vibration

Table 5 demonstrated and distribution of the FTIR spectrum analysis of PVAc. When Absorption were O=C=O, Band intensity wavenumber were weak, Wave number were 2343 and Assignment were Stretching vibration. When Absorption were C-H, Band intensity wavenumber were Medium, Wave number were 1365 and Assignment were Bending vibration. When Absorption were C-C, Band intensity wavenumber were strong, Wave number were 1023 and Assignment were Stretching vibration.

Table 6: Tensile strength and elongation (%) of natural rubber (NR), polyvinyl acetate (PVAc), NR/PVAc blend, ONR/PVAc blend, NR60/PVAc40 blend-based nano-cellulose (5%) composite and ONR60/PVVc40 blend-based nano-cellulose (5%) composite

Materials	**Tensile strength (N/mm^2)**	**Elongation (%)**
Natural rubber	0.27	67.96
NR60/PVAc40	3.31	42.2
NR40/PVAc60	7.76	11.77
ONR60/PVAc40	1.71	71
ONR40/PVAc60	3.52	8.2
NR60/PVAc40/NC	0.92	12.3
ONR60/PVAc40/NC	5.69	5
PVAc	13.23	3.55

Table 6 demonstrated and distribution of the Tensile strength and elongation (%) of natural rubber (NR), polyvinyl acetate (PVAc), NR/PVAc blend, ONR/PVAc blend, NR60/PVAc40 blend-based nano-cellulose (5%) composite and ONR60/PVVc40 blend-based nano-cellulose (5%) composite. Tensile strength of Natural rubber, NR60/PVAc40, ONR60/PVAc40, ONR40/PVAc60, NR60/PVAc40/NC, ONR60/PVAc40/NC, PVAc were 0.27, 3.31, 7.76, 1.71, 3.52, 0.92, 5.69, 13.23 and Elongation were 67.96, 42.2, 11.77, 71, 8.2, 12.3, 5, 3.55 respectively.

DISCUSSION

A substitute route for the in-situ instruction of bacterial cellulose-based nanocomposites entails the bacterial cellulose reducing groups that can entrap titanium dioxide nanoparticles inside the fibers net. In this research the technique indicates the advantage of the exterior chemical that are no longer brought to the reactant mixture and for that reason avoids adventitious contaminations that may additionally intervene in some applications [8]. In sol gel method, titanium dioxide is immobilized with the aid of 3-Glycidyloxy propyl trimethoxy silane. After application of the two strategies on the different textile fabric substrates the characterization of the handled fabric has verified a comparative distinction between these two methods.

In our study, according to Constituent, Cellulose was 43.4822%, Alpha-Cellulose were 34.8427% and Cellulose were 8.6395%. And according to Cellulose content in other plant straws, Rice Straw were 32.15%, Sugarcane were 41-43%, Rye Straw were 31.8-42.64%, Corn Stalks were 29.80% and Wheat Straw were 34-40%.

It is really worth to notice that at some stage in biosynthesis, the packaging technique leads to relatively pure and crystalline cellulose. BC has been said to exhibit greater crystallinity than 80% whilst cellulose from plant life incorporates extra amorphous areas and shows crystallinity values ranging from 40–85% [9]. In phrases of crystallinity, cellulose shows six exclusive polymorphs, namely, I, II, IIII, IIIII, IVI and IVII [10]. Cellulose type I is the most plentiful structure in nature and the one predominantly produced by bacteria [11]. The crystalline shape of cellulose I is a combination of two awesome crystalline structure forms: cellulose Ia (triclinic) and cellulose Ib (monoclinic). Depending on the supply of the cellulose, the relative abundance ratio of these two allomorphs varies. Cellulose Ib is the principal constituent of the cellulose from plants, whilst bacterial cellulose is prosperous in the Ia allomorph [12]. The relative heights of these peaks fluctuate markedly from these on perfect diffraction patterns or these from cellulose in plant cell partitions due to the fact of desired orientation of the crystallites to the X-ray beam [13].

In our present study, according to FTIR spectrum analysis of NR, Absorption were C-H, Band intensity wavenumber were Strong, Wave number were 2957 and Assignment were Stretching vibration. When Absorption were CH, Band intensity wavenumber were Strong, Wave number were 2929 and Assignment were Stretching vibration. When Absorption were C=C, Band intensity wavenumber were Medium, Wave number were 1644 and Assignment were Stretching vibration. And according to FTIR spectrum analysis of oxidized natural rubber, Absorption were O-H, Band intensity wavenumber were Strong, Wave number were 3200 and Assignment were Stretching vibration. When Absorption were O=C=O, Band intensity wavenumber were Medium, Wave number were 2343 and Assignment were Stretching vibration. When Absorption were C=O, Band intensity wavenumber were Strong, Wave number were 1715 and Assignment were Stretching vibration. When Absorption were C-H, Band intensity wavenumber were Medium, Wave number were 1447 and Assignment were C-H bending.

Due to this excessive crystallinity, presence of several strong hydrogen bonds and its fiber conformation, BC indicates gorgeous mechanical overall performance even in wet state. For BC fibers, values from 10–65 GPa and 80–800 MPa have been pronounced for young's modulus and tensile strength, respectively [14, 15]. These values commonly range relying on the bacterial strain, cultivation method, processing of BC and water content material (which has plastifying effect). In fact, BC has a robust affinity with water, so in the swollen nation values of 8 MPa and 1.5 MPa of Young's modulus and tensile strength, respectively, can be located [16]. Indeed, BC can be viewed a hydrogel with an excessive water keeping ability which can attain one hundred instances its very own weight. Moreover, BC is viewed a biodegradable material given that the hydrophilicity and water uptake functionality are elements associated to the biodegradability. Hydrophilic substances are extra inclined to hydrolysis which is a system that initiates the degradation of polymers, and approves the materials to be colonized by means of microorganisms and fungi, which will have got admission to carbon as food [17]. Due to this feature, the application of BC for the manufacturing of biodegradable packaging substances has been investigated [18, 19]. In addition to these fascinating residences and conformation, BC has demonstrated to be biocompatible in animal research [20, 21]. For profitable organic implants, the biomaterial ought to set off cellular expansion, integration and interactions with biological tissues, and it has been established that biological cells can anchor, scatter and combine with bacterial cellulose except poisonous response, opening new purposes in the biomedical area such as tissue engineering and regenerative medicine. Some of the purposes associated to this place encompass improvement of artificial blood vessels, artificial skin, biosensors, dental implants, warmness valves and bone regeneration [22, 23].

Our study demonstrated and distribution of the FTIR spectrum analysis of PVAc. When Absorption were O=C=O, Band intensity wavenumber were weak, Wave number were 2343 and Assignment were Stretching vibration. When Absorption were C-H, Band intensity wavenumber were Medium, Wave number were 1365 and Assignment were Bending vibration. When Absorption were C-C, Band intensity wavenumber were strong, Wave number were 1023 and Assignment were Stretching vibration. And also demonstrated and distribution of the Tensile strength and elongation (%) of natural rubber (NR), polyvinyl acetate (PVAc), NR/PVAc blend, ONR/PVAc blend, NR60/PVAc40 blend-based nano-cellulose (5%) composite and ONR60/PVVc40 blend-based nano-cellulose (5%) composite. Tensile strength of Natural rubber, NR60/PVAc40, ONR60/PVAc40, ONR40/PVAc60, NR60/PVAc40/NC, ONR60/PVAc40/NC, PVAc were 0.27, 3.31, 7.76, 1.71, 3.52, 0.92, 5.69, 13.23 and Elongation were 67.96, 42.2, 11.77, 71, 8.2, 12.3, 5, 3.55 respectively.

Moreover, this biopolymer indicates a huge chemical and bodily modifiability. BC membranes possess free hydroxyl agencies on the surface, so they can be without problems modified with different polymers or components to attain distinctive surface characteristics such as lipophilic or hydrophilic, magnetic and optical properties [24, 25]. Besides, BC has the opportunity to be used as matrix or reinforcement of composites and hybrid substances organized via ex situ and in situ methods. Ex situ strategies consist on the aggregate of the purified BC by using the impregnation approach to fix, coat or infiltrate the additive/polymer in the areas between the nanofibers of the BC membrane. Using in situ methods, BC-based composites can be got by means of the addition of some components or soluble polymers to the tradition medium so that the additive is included into the BC developing nanofiber community all through its biosynthesis. Both education techniques lead to BC with distinctive residences and conformations [26-28]. Taking into account all these features, it is now not stunning that its manufacturing and mixture or amendment with different substances for the improvement of hybrid units and composites have been considerably studied in recent decades for many different applications.

CONCLUSION

Novel eco-friendly bio-nano-composites substances primarily based on renewable and sustainable assets namely natural rubber (NR) and nano-cellulose received from rapeseed plant straw as properly as poly vinyl acetate (PVAc) have been developed. Preparation of nano-cellulose from rapeseed plant straw, synthesis of poly vinyl acetate, synthesis of oxidized natural rubber (ONR), processing of NR/PVAc and ONR/PVAc blends, processing of NR/PVAc and ONR/PVAc blend-based nano-cellulose (5%) composites as properly as their characterization have been carried out. Natural rubber and poly vinyl acetate flims have been received by using casting and evaporating of water from natural rubber latex and PVAc emulsion respectively. NR/PVAc and ONR/PVAc blends of unique ratios have been organized through simple blending and accompanied by using evaporating of the blends. Nano-composite films had been additionally received by way of casting after evaporating a combination of natural rubber (NR) latex, PVAc emulsion and nano-cellulose (NC) suspension. In order to enhance the interfacial interactions amongst the composite factors such as NR, PVAc and NC, oxidation of the NR latex was once carried out by way of KMnO4 before blending mixing with PVAc emulsion and NC suspension.

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