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Extraction of Nano-cellulose from Banana Rachis (Agro-waste) and Preparation of Nanocellulose-Clay Nanofilter for the Industrial Wastewater Purification

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ABSTRACT

Industrial dye wastes represent one of the most problematic groups of pollutants because they can be easily identified by the human naked eye and are not easily biodegradable. This research article highlights some recent development of Nanocellulose-Clay Nanofilter (NCCNF) in water treatment technologies. Nanocellulose (NC) was extracted for this research work from banana tree rachis fiber (*Ebelmuschus esculentus L*). Firstly, obtained raw fibers from banana rachis were treated with soap solution and benzene-alcohol (1:2) mixture then alkali wash with 17.5% NaOH solution andfinally, bleachingaswellas60%sulphuric acid(H2SO4) hydrolysis wascarried outonitsuccessively. BythiswayNCwasextractedsuccessfullyfromrawrachisfiber.Ontheotherhand,collectedwhiteclaytreatedwith ethylene diamine (5%). In this work, we described briefly how NC was produced and its peripheral surfaces were activated for high adsorption. The anti-fouling properties of 'NC-Clay' based nano-filters will be also highlighted. However, NC-Clay Nanofilter (NCCNF) was prepared by solution casting method. And the samples i,e RF, ATF, BF, NCand NCCNFwere characterized by Fourier transforms infrared spectroscopy (FTIR), Thermo gravimetric analysis (TGA), X-ray diffraction (X-RD), Scanning electron microscopy (SEM) analysis. Analysis data supports this bio nanofilter is highly crystalline, thermally stable, have good surface morphology, and also have strong composite forming capacity as well as biodegradable. On the other hand waste water containing heavy metal solutions were also characterized by UV-Visible and AAStechniques.

Keywords: Nano-cellulose; Clay; Heavy metal; Agro-waste; Waste waters; Water purification.

Introduction

As an agricultural country in Bangladesh every year a huge amount of banana rachis (Figure 1) are expelled out from the banana production and processing farming land/zone as an agro waste materials which causes serious pollution in our environment. We know that banana rachis is a good source of cellulose. This is why for the extraction of nano-cellulose banana rachis (*Ebelmuschus esculentus L*) was chosen as a source material of cellulose in this research work. Here, banana rachis was chosen because they were agricultural waste material thus they were cheap, edible, non-toxic as well as biodegradable i,e., eco-friendly. However, cellulose referred as a tough, fibrous, linear syndiotactic homo polymer which composed of D-anhydro glucopyranose units which are linked by β-(1,4)-glycosidic bonds [1]. They are undoubtedly the most abundant natural biopolymer on the earth, widely subsistent over a diversity of origin for example marine animals, bacterial and plants. With the growth of the environmental awareness we are looking for eco-friendly materials to eliminate the fossil fuel based polymeric materials, in order to preserve and protect our environment [2]. Due to biodegradability and biocompatibility NC has an important application on the field of nanofilter, nanocomposites and bio-nanomedicine. In some cases, the nanostructured form of cellulose nano crystals (CNC) was produce by the interaction of bacteria, also known as bacterial Nano-cellulose [3].

On contrast clay materials are the combinations of solid, fluids and gasious phases [4]. The solid phases are of mineral and organic substances that make up the crystal geometrical structure of clay complex [5-6]. Chemically clays are plastic due to their water content and become hard, brittle and non-plastic upon drying or firing [7]. Besides this clays have an extraordinary water/moisture absorption capacity due to its phyllosilicate minerals. Because phyllosilicate minerals always help it to trap a variable amounts of water into the mineral structure. For this absorption ability it was chosen and modified by 5% ethylene diamine with a view to enhance its hydrophilic nature as well as surface activity. However, Studies in the early twenty first century have inspected that the absorption capacities of clay in various applications, as for examples the separation of toxic heavy metals, dye molecules as well as hazardous organic or inorganic substances from waste water and air purification [8-9]. Depending upon the soil's content in which it is found, clay can appear in various colours such as dull grey to white and brownish to deep orange-red. But here to fabricate NCCNF we used partially white clay as a filler material.

Figure 1: Banana rachis (agro-waste)

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However, there were many processes for the treatment of industrial waste water for example biological treatment, catalytic oxidation, filtration, sorption process etc. But here we used nano-filtration process for the purification of industrial waste water. We produced NC-clay composites as a nanofilter, it was chosen because of its bio-degradability (both the reinforcement and the matrix were bio based). NCCNF composite is better than any other filter composite. The most important cause to choose CNC-clay composites that the main raw material (NC) was collected from agricultural wastage (a bio-resource), so it is cheaper, biodegradable, nontoxic and environmental friendly nanocomposite. They also have thermal stability, high crystallinity, high composite forming ability, good surface activity and have better abrasive properties. On contrast, clays are highly crystalline and they have a good absorption capacity against important water pollutants, e.g., heavy metal species, dye wastes and organic molecules. So, CNC-clay composites are very effective for the purification of waste water. In this research work, it was tried to find out how to use properly the agro wastage for the waste water treatment technology in very economical, effectively as well as easy way.

Materials and Methods

Materials

Banana (*Ebelmuschus esculentus L*) rachis fibre was the raw material of NC which collected from banana cultivating and processing landing area near Islamic University, Kushtia and white clays were collected from Vejaypur, Bangladesh. Sodium Hydroxide (NaOH) 98% pure, Sulfuric acid (H2SO4) 98% pure, Sodium acetate (CH3COONa) 98% pure, Glacial acetic acid (CH3COOH) 100% pure, Sodium chlorite (NaClO2) 80% pure, Sodium meta-bi-sulphite (Na2S2O5) 99.9% pure, 5% ethylene diamine, theses all were from BDH in England and sands were taken from local area, 0.0025% solution of direct yellow (vat dye) origin China, copper sulphate (CuSO4) and nickel sulphate (NiSO4) from Indian origin and 10% soap solution from local market.

Methods

Extraction of nano-cellulose (NC) from banana rachis fibers: The Raw Fiber (RF) (Figure 2) was collected from banana (*Ebelmuschus esculentus L*) rachis, when banana rachis got mature then it was retted into a local canal for 25-30 days, after bacterial decomposition the RF was taken out from banana rachis. Than it was washed in clean water for several times and dried in air without exposing sunlight. Finally, the RF was dried in an electric oven at 105°C and stored in an air tight container. This fiber was applied for various medicinal uses [10]. The removal of impurities such as dirty materials, fatty, waxy and gummy substances from fiber materials is called scouring. It is carried out by the use of 10% soap solutions/detergents. The ratio of the RF to solution was 1:50. It was conducting for 2 hours at 40°C. Then the fiber was thoroughly washed with distilled water for several times and dried at 80°C-90°C for 24 hours. Then it was treated with 17.5% NaOH solution at 500C temperature for 4 hours with constant stirring. This is called alkali treatment, the ratio of fiber and solution was 1:25. Then the Alkali Treated Fiber (ATF) was obtained (Figure 3). Then the ATF was treated with 0.7% NaClO2 at 90°C for 2 hours with occasional steering [11] to perform bleaching operation. The obtained yield was washed several times with DI water to remove any NaClO2 sticking on the spaceman surface. A final pH of 4 was maintained, the sample were then dried at 100°C for 6 hours. By this way Bleached Fiber (BF) ware obtained (Figure 4). However, to produce NC, firstly the BF was cutting into as small as possible size then, NC was prepared by the double-acid hydrolysis. The hydrolysis was carried out with H2SO4 solution (60 wt. %) at 45°C under magnetic stirrer with hot plate. The BF to liquor ratio was 1:10. After 30 minutes, when the colour appeared to brownish the hydrolysis was stopped by adding 6 fold excess ice cool water into the reaction vessel and simultaneously the magnetic stirring was running for next 30 minutes again. After completing reaction the resulting mixture was cooled down at 20°C and centrifuged. The solid fraction was washed out by DI water until to obtain a neutral pH. Finally, the NC suspension was obtained. The newly generated suspension was dispersed into "ethanol or absolute alcohol" into a safe as well as clean glass vessel (Figure 5).

Figure 2: Raw fiber

Figure 3: Fiber during alkali treatment

Figure 4: Bleached fiber

Figure 5: Nano cellulosic fiber

Fabrication of NCCNF: Many types of blending methods were followed for the preparation of various polymeric blends and composites such as melting process, molding process, solution casting process etc. But solution casting process is the most applied methods for preparing NCCNF composites. To fabricate it first of all collected white clay (Figure 6) was dissolved separately in 5% diethyl amine solvents for 48 hours. Then the solution was allowed to keep for filtration by a suitable filter cloth. After completing filtration the filtrate part was sent to an electrical oven for drying at 105°C. Before fabricating NCCNF composite, the modified clay and NC were mixed with each other into a mixing tank at the ratio of 40:60 in which 40% of NC and 60% of clay. To produce a homogenous mixture it was blended well into the matrix of absolute alcohol.

Figure 6: Collected white clay

Preparation of waste water solution: For preparing 0.025% solution of direct yellow, at first 0.025 gm. of dye was taken and kept in a glass beaker which contains 1000 ml of DI water. Then 0.025 gm. of copper sulphate ($CuSO_4$) and 0.025 gm. of nickel sulphate ($NiSO_4$) (water soluble) were also taken into the same beaker. It was shown in Figure 07.

Figure 7: Waste water solution

Column preparation: At first a filter column was selected which contain a 0.5 cm cotton bed at the lower part (Figure 8). Then a sand layer was placed which was 2.0 cm thick. After that the NCCNF composite layer (2.50 cm thick) was introduced into the glass filter column. The total thickness of the NCCNF composite should not be higher than 5.00 cm which contain 60% modified clay and 40% NC. Because when the thickness exceeds that limit then it would take a huge amount of time to pass the water through the filter layer as well as if the layer was so thin from this desired level then a serious problem might be occurred i.e, the filter layer would lose the resistance properties and allowed to pass all the dye as well as heavy metal molecules through it.

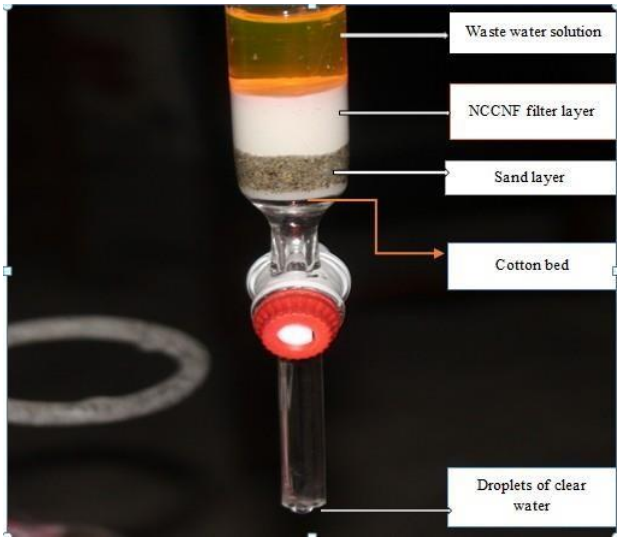

Figure 8: NCCNF nanofilter during waste water purification

Filtration of industrial waste water: To administrate filtration of the supplied waste water the prepared column need to keep vertical position. Then 0.025% dye solution containing heavy metal solution was poured into the standing column which was ready for waste water treatment and recording the time until the whole water passed through the filter layer. If there was any air space or any microscopic gap present in the NCCNF layer then the dye molecules might also be passed with the water through it. So it was very important to handle the Nanofilter carefully and protect the perforation or microscopic gaps of NCCNF filter. Here, for passing the nano-filter layer, 20 ml waste water solution took the time which was around 10 hours. Finally, the filtrate water was found with a clear appearance (Figure 8), *i.e.* no dye molecules were present or visible, and this was very optimistic and positive matter of this research. However, the polymeric samples and water samples were characterized by FTIR, SEM, XRD, TGA, UV-visible and AAS techniques.

FTIR analysis: FTIR technique is usually applied to investigate intermolecular and intra-molecular hydrogen bonding of different polymeric spacemen because it is the most frequent technique to observe inter-molecular and intra-molecular interaction in polymeric compounds and composites. Prior to examined by FTIR (Shimadzu IR Prestige-21 spectrometer), the samples were washed according to

the particular procedure.

Scanning electron microscope

To characterize the surface morphology of polymeric samples scanning electron microscopy (SEM) technique is generally conducted. The specimens were sputter coated with gold to protect charging. The micrographs were taken at a magnification of 100, 500 and 10,000 x. The surface morphology of RF, ATF, BF, NC and NCCNF nanofilter composite were examined by scanning electron microscope (FEI QUANTA 200 3D) with an accelerating voltage 10 KV.

X-ray diffraction

X-ray diffraction (XRD) technique was applied to detect the crystallinity of the supplied polymeric samples. The structure has been observed by DFT (density functional calculation) Nikolovet al [12], Minke and Blackwell [13] on basis of the intensity data. The crystalline structure of RF, ATF, BF, NC and NCCNF nanofilter composite were evaluated by a WAXD (BRUKER D8 ADVANCE wide angle) diffractometer using Cu Kα radiation (α=0.154 nm) voltage of 50 KV and current of 40 mA with 2Θ ranges from 100 to 800 increase in steps of 5.020/min.

TGA analysis

To evaluate the thermal stability due to decomposition of different polymeric compounds Thermo Gravimetric Analysis (TGA) technique is normally administrated [14-16]. The thermal stability of RF, ATF, BF, NC and NCCNF composites were investigated by the Thermal gravimetric analyser (TGA 6300 Seiko Instrument, Japan). About 25 mg of each specimen were taken for observation. The samples were heated up steadily at a rate of 20°C/min from 26 to 600°C under continuous flow of nitrogen at 60 ml/min. To get accurate result, analysis was carried out more than one times for each sample.

AAS analysis

Atomic Absorption Spectroscopy (AAS) is a spectra analytical procedure for the quantitative determination of chemical elements using the absorption of light by free atoms in the excited state [17]. The amount of heavy metal which was present in the supplied waste water solution before and after purification (filtration) were measured by atomic absorption spectroscopy (AAS) Varian, spectra AA 220FS with direct flame method.

UV-vis spectroscopy

Ultraviolet-visible spectrophotometry (UV-Vis) refers to absorption spectroscopy or reflectance spectroscopy in the UV spectral region which uses the visible and adjacent light. In this optical range of the electromagnetic spectrum, atoms and molecules undergo electronic transitions [18]. The percentage of dye waste which was also present in the supplied waste water solution before and after purification (filtration) was measured by UV-VIS, V-1200, UV-1600PC spectrophotometry.

Result and Discussions

FTIR spectroscopy analysis

The structural changes in chemical composition were examined by FTIR spectroscopy. Figure 9 shows the FTIR spectra of RF, ATF, BF, NC and NCCNF composites. Noteworthy that there was no obvious difference in the spectra of first four samples. This result indicates that there was no chemical changes take place as well as no new substance was produced during the chemical reaction. ATF, BF, NC as well as NCCNF composites were shifted slightly (Table 1). The BF spectrum showed distinctive pecks and bands for carbohydrates (associated with it), a small peak in the range of 3300-3400 cm^{-1} with board O-H stretching was observed in NCCNF composite for overlapping with N-H bond str. [19]. The C-H stretching peaks were shifted in higher wave number 2950 cm^{-1} to 2880 cm^{-1} after adding hydroxyl group of BF. On contrast, C=O stretching in ATF shifted to NCCNF from region of 1741.72 $cm^{-1}$to 1701.22 cm^{-1} in C [20]. It referred that NC and modified clay produced intermolecular hydrogen bonding between O-H, C=O, group to weaken the C=O in ester group in clay. Similarly, other bending peak shifted to the higher wave number for intermolecular bond formation. Moreover, Table 2 showed that free –OH group,–OH out of plane bending vibration and O=N=O bending vibration peaks were appeared at the region of 3695.61-3620.39 cm^{-1},786.92-692.44 cm^{-1}, 583-468.70 cm^{-1} respectively in NCCNF composites only for modified clay because they were totally absent in all RF, ATF, BF and NC. However, most of the characteristic absorption peak of the NCCNF composite shifted to lower wave number with increasing peak fronting. This information could provide a confirmation that NC and Clay didn't have better compatibility in composites via intermolecular hydrogen bonding and this supports previously result.

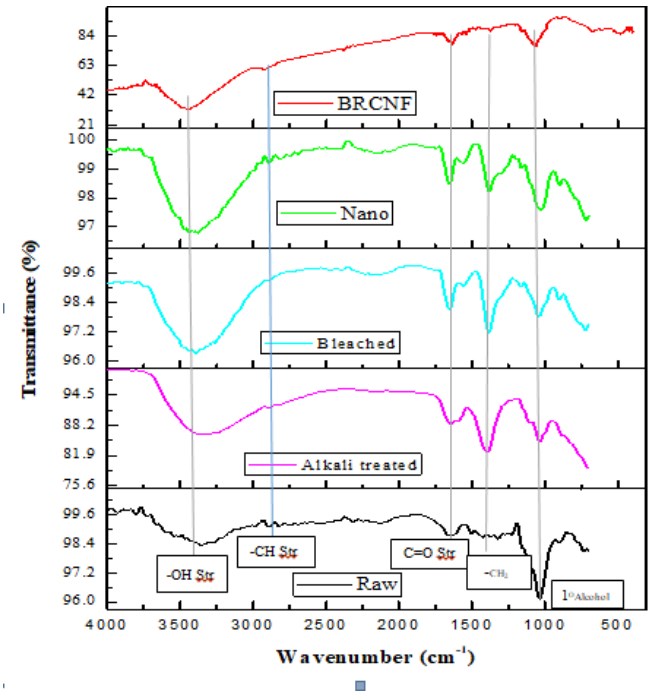

Figure 9: FTIR spectra of RF, ATF, BF, NC and NCCNF nanofilter.

X-ray diffraction analysis

The RF, ATF and BF were characterized by XRD to check the crystallinity, and the X-ray diffractographs were shown in Figure 10. While, in case each diffractograph exhibited two diffraction peaks at near 2θ=22.5o and from 15o to 18o, this is why, it showed small stereo crystallinity. On contrast, X-RD analysis of NC shows a sharp peak at 2θ=2°C and small peak at 22.5°C (Figure 10). For this reason, NC exhibits semi-crystallinity. The crystallinity index (CrI) of RF, ATF, BF and NC was 52.08%, 61.69%, 59.50% and 69.26% respectively.

So, it can be concluded that after chemical treatment the degree of crystallinity of fiber were increased.

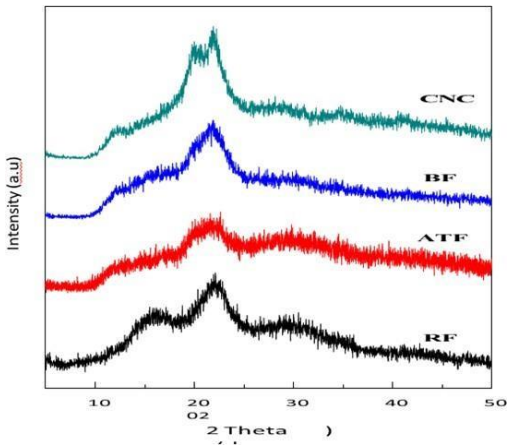

Figure 10: XRD curve of RF, ATF, BF and NC fiber

The degree of crystallinity was calculated according to the Segal method [21]. Crystallinity index (CrI) was calculated according to the following equation-

$$CrI=(I_{002} - I_{am})/I_{002} \times 100\%$$

Where, I_{002} is the highest of the diffraction intensity (the 002 plane), and I_{am} is the lowest diffraction intensity (between 101 and 002 peaks).

On the other hand, Figure 11 Represent the X-ray diffractograph of NCCNF composite. Here, the crystallinity Index (CrI) was 91.17%. It showed a sharp and broad peak at 2θ=12o and 19o correspond to the homo-crystalline structure, the 2θ=20.4 and 26.5 were more remarkable peaks showing the crystallinity of the cellulose reinforcement by clay. From the above analysis, it was clear that, fiber and NC were semi-crystalline in nature. But, after the addition of modified clay, the NCCNF composite exhibits highly crystalline character. This was the fact that addition of modified clay acts as nucleating agents to accelerate the crystallization of NCCNF composite [22].

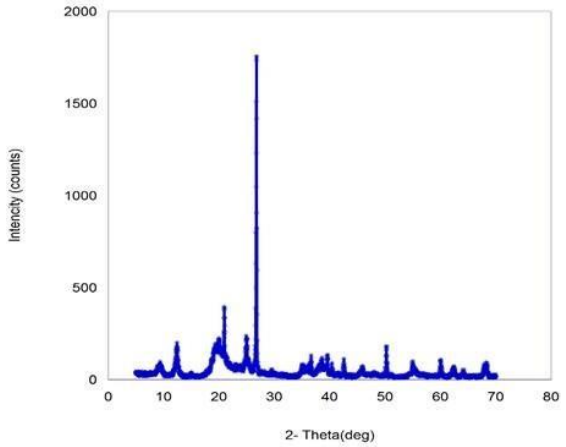

Figure 11: X-RD analysis of NCCNF nanofilter.

SEM analysis

The SEM micrograph of RF, BF, NC and NCCNF composite were shows in Figures 12. Here, RF (Figure 12A) clearly demonstrated the presence of longitudinally oriented unit cells. The intercellular gap was filled up by the lignin and fatty substances, which hold the unit cells firmly in a polymeric fiber. In Figure 12B showed the comparative SEM picture. From this figure the drastic difference in the surface morphology between RF and BF could be easily identified. Generally, natural cellulosic fiber was negatively charged due to the presence of carboxyl and hydroxyl groups as mentioned in the literature [23]. In case of RF, these groups were covered by lignin, present in the primary cell wall of the fiber shown in Figure 12B. Unlike the RF the surface of BF seems to be free from surface debris and overgrowth. This was undoubtedly due to removal of surface impurities as well as lignin and hemi-cellulose from it. The NC appeared in Figure 12C, micro and nanoparticle-like structure, it could be seen that the NC had the diameter at a range of around 400-800 nm with some micro-cellulose. Noteworthy that in the micrograph of NC there was a lot of honey comb structures appeared which is very effective for producing any types of strong polymeric nano-composites. Because, this structures of NC fiber help to interlock the filler materials when they combined with each other. In Figure 12D showed that SEM picture of NCCNF composites. Comparison of the micrographs of the RF, BF and NC showed that a considerable quantity of modified clay was oriented onto NC surface. The intercellular gaps were also decreased due to the deposition of clay as fillers. As the surface was covered with polymer, it became more or less uniform and smooths [22-24].

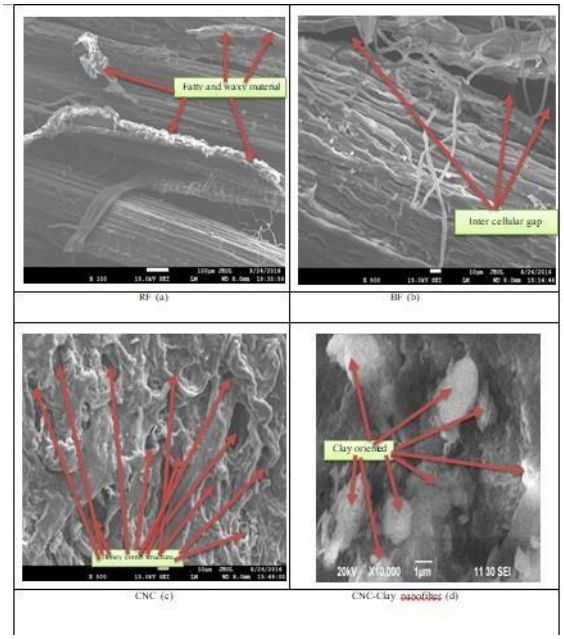

Figure 12: SEM micrograph for surface morphology analysis of RF, BF, NC as well as NCCNF nanofilter 10,000×.

TGA analysis

TGA, curves were represented and discussed below: In Figure 13 the TGA showed the initial loss of 12.1% due to the presence of internal water. Then the loss of weight was initial slower rate and

ending rapid rate. The onset temperature, and the highest degradation temperature of RF, ATF, BF, NC were at 270°C, 265°C, 215°C and 388°C, 378°C, 365°C respectively (Table 3). The main decomposition at this temperature was due to depolymerization, dehydration, and decomposition of hydroxyl units in polymeric association. The total degradation of RF, ATF, BF, NC were 68.3%, 69.8%, and 55.2% respectively. On the other hand, Figure 14 showed that the thermal status of NCCNF composite. Where at 22.7°C there was no loss but after sometime at 69.5°C the loss rate was very high *i.e* about 37.11%. At the time 9.70 min as well as 118.3°C temperature the decomposition was almost 89.11%. But after that stage the degradation became constant such as at 373.24°C and 599.68°C the decomposition were 91.84% and 92.50% respectively (Table 3).

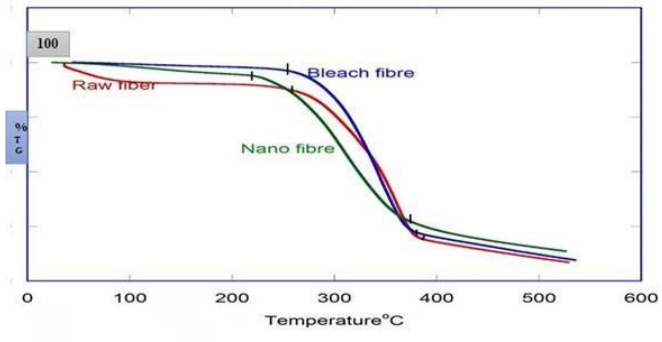

Figure 13: comparative TGA curve of RF, BF and NC fiber

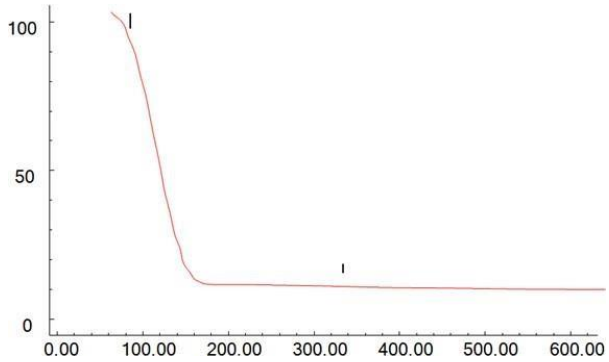

Figure 14: TGA curve of NCCNF nanofilter composite

UV-visible spectroscopy analysis

Figure 15 showed the Comparative line graph of UV analysis. Where, Blue colour represented waste water solution before filtration and Black colour represented the solution after filtration (by NCCNF nanofilter). From the diagram we could clearly see that the λ_{max} was appeared for the waste water sample at 517 nm which was responsible for direct yellow (a vat dye) [29]. On the other hand, after filtration there was no absorption maximum (λ_{max}) at this region for the filtrate water or purified solution. So, it can be concluded that there was no dye present in the purified solution, all dye molecules were absorbed by NCCNF nanofilter.

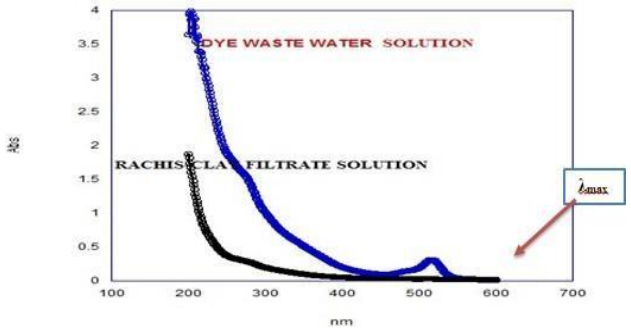

Figure 15: UV analysis of dye containing in waste waters before and after filtration.

Atomic absorption spectroscopy analysis (AAS)

Figure 16 Showed the data of AAS analysis for Ni and Cu were present in waste water before and after filtration. Where, S1: Represented the supplied waste water sample before filtration and S2: Represented after filtration. Here from Table 2 we could clearly understand that the purification of nickel was 78.18%. On the other hand, the purification of copper from waste water sample was very remarkable *i.e*, the presentence of copper purification was Below the Detection Limit (BDL) *i.e* 98.74% which was outstanding. So, from the above analysis we can summarized that the NCCNF is better for removing of Cu and Ni from the industrial waste water.

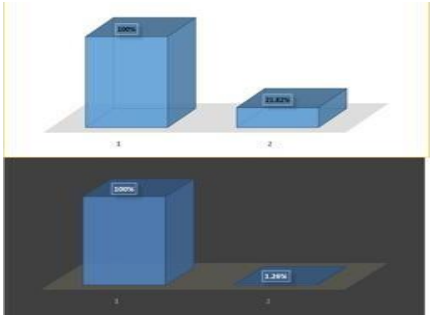

Figure 16: AAS analysis for "Ni" and "Cu" containing in waste water before and after filtration.

Conclusion

In a nut shell, we can draw a conclusion that for the purification of industrial waste water NCCNF played an important role. This biodegradable bio-nanofilter was produced due to water purification purposes. Because we know that today, we face a global water crisis. This global water challenge will become a greater problem in between the next 30 years, because of the demands on water sources will continue to dilate for domestic, agriculture, industrial application. So, waste water purification is one of the most important challenges for the next generation especially for the industrial sector. This will require sustainable and economic water treatment technology. As banana rachis fibers and clays are cheaper, nontoxic, biodegradable, biocompatible and environmental friendly, these bio nano-composites may have a great application in industrial waste water treatment technology. The new class of renewable bio-nanofilter composites

showed remarkable progress in mechanical and barrier properties during filtration of industrial effluent compared to others because about all the dye molecules, 98.74 % of Cu and 78.18% Ni (heavy metals) were purified in this experiment. Thus NCCNF can capture the new market in water treatment application.

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Declaration

There are no conflicts of interest regarding this research work.

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