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Production of Nanocellulose Filter Paper For Water Purification From Malaysian Sustainable Forest Resources (Shorea Roxburghii) Through High-Performance Filtration in Industrial Wastewater Treatment

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Abstract: Nanostructured cellulose has received enormous attention due to its unique properties. Being the most abundant organic polymer on earth, cellulose is the most suitable candidate for alternative replacement of petroleum-based products. Therefore, this study aims to identify the effectiveness of filter paper using renewable forest resources and the advances made in nanotechnology, especially cellulosic nanomaterials, to develop novel or superior products with enhanced performance attributes. Shorea roxburghii was used as a raw material for the production of nanocellulose filter paper intended use in water purification for the dye removal in textile wastewater. Reactive dyes were considered to be the most problematic compound in textile wastewater due to their high water solubility, high stability and persistence in nature. This study was carried out for the utilization of nanofibrillated cellulose (NFC) filter paper as adsorbent for the removal of dyes from wastewater and to establish it as a standard wastewater treatment process for composite textile industry. Based on the researcher's study on nanocellulose filter paper, this absorbent was capable of being used as a dye removal.

Keywords: Nanocellulose, Dye wastewater, Filter paper

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1. Introduction

Textile industries use large quantities of water and chemicals for processes of finishing and dyeing. Due to the persistent and recalcitrant nature of different dyes, the release of dyes into waste water by different industries poses serious environmental problems. Some of the dye from the textile wastewater industry are made of hazardous chemicals such as benzidine and metals that are carcinogenic and mutagenic to all life forms [1]. Dye waste water is usually made up of a variety of chemicals including acids, bases, detergents, soluble solids, toxic compounds and colour. Colour is the most visible contaminant even at very low concentrations and must be eliminated or decoloured before the waste water can be discharged. Not only does the inclusion of colours in water resources make them aesthetically undesirable, but it also raises serious health risk factors for living organisms and the environment. Therefore, the wastewater needs to be treated prior to discharge by removing dye color in order to protect environment and as per the statutory guidelines.

Shorea roxburghii or knows as Meranti Temak Nipis in malays is a rain forest species that belongs to the Dipterocarpaceae white meranti timber group, categorized as lightweight hardwood plant. This species in the tropics is useful for forestry due to its tolerance to high survival and fire heavy drought after the forest fire [2]. Shorea roxburghii consists of approximately 194 species, and 163 of it is occur in Malaysia. The cellulose content of Shorea roxburghii is greater than the required amount. In industries such as pulp, paper and wood hydrolysis the cellulose contents of a plant material are important as it can affects the quality of the products [3]. Nanofibrillated cellulose (NFC) was prepared from Shorea roxburghii wood pulp via 64 wt% sulfuric acid hydrolysis. Prior to acid hydrolysis, Shorea roxburghii was subjected to pulping followed by bleaching in order to remove non-cellulosic fragments. Grinding as the most common technique used for mechanical NFC production was carried out to produce the nanofibril bundles with diameters between 6 and 100 nm [4].

2. Literature Review

The textile industry in Malaysia is well known for a wide variety of activities, including polymerization and man-made fiber processing, spinning, weaving, knitting, pattern, dyeing and printing. In every stage of textile industry various types of dyes are used to color their products [23]. Since the textile industry uses high quantities of chemicals and water to form waste after processing, this sector has been convicted of pollution as one of the largest criminals in the world [5]. Additionally, the formation of poisonous carcinogens is a major problem when the powdered dye is released directly into municipal and municipal wastewater plants [6] . Due to its wide coverage of pH, COD, dissolved solids and a variety of synthetic dyes, the treatment of textile industry wastewater remains a major challenge. Textile wastewater has a pH range of 2 to 14, a COD range of 50 to 18,000 mg / L and a total dissolved solid (TDS) range of 50 to 6000 mg / L. The wide variation in the characteristics of textile wastewater is due to the large number of chemical components used in textile manufacturing [7]. As the issue of the environment has become a global concern, industrial effluent must be treated before it is released into the environment.

The color removal was extensively studied using different methods such as coagulation, flocculation, ultra-filtration, nano-filtration, photo oxidation, activated carbon etc. However, the use of these methods is limited by high maintenance, suitable facilities are required and it is quite expensive. The removal of dyes from wastewater using adsorption process by agricultural waste provides an alternative treatment. It is an efficient method of eliminating different contaminants from aqueous solution and is also one of the most cost-effective alternative methods of handling dye waste. Adequate to simple nature, adsorption has advantages over other method and can involve low investment in terms of both initial and land required.

Nowadays, adsorption removal of dyes has been shown to be an effective way to treat dye-containing effluent [9]. The adsorption process refers to the gradual transition of the dye from the solution to the adsorbent surface reaching a point after which the dye distribution in the solution remains unchanged between the adsorbent. Membrane, combined with adsorption and membrane isolation, is one of the important techniques of wastewater treatment [10]. Filter paper, a typical cellulose membrane, was usually chosen to be conventional filter material due to its low cost, non-toxic and mature production process. The functional groups on the filter paper surface can greatly enhance the material's adsorption efficiency after alteration [11]. This study was carried out for the utilization of nanofibrillated cellulose (NFC) filter paper as adsorbent for the removal of dyes from wastewater and to establish it as a standard wastewater treatment process for composite textile industry.

Cellulose is the most abundant polymer in nature and originates mostly from trees and plants cell walls. Wood, which contains about 40.00 % cellulose, has remained the major source of cellulose in the form of wood pulp, accounting for 90.00 % of global production of pulp. Nanofibrillated Cellulose as a new cellulosic material was introduced by Turbak and Herrick who produced cellulose with lateral dimensions in nanometres range by passing a softwood pulp aqueous suspension several times through a high-pressure homogenizer [12-13]. Besides the availability of cellulose worldwide, strong interest in NFCs is also because of increased demand for biodegradable and sustainable products and also due to its exceptional characteristics that offers vast potential across many application fields. Some of NFC that has been extracted from various resources include bleached hardwood pulp. NFCs can be further divided into three categories; nanofibrillated cellulose (NFC), cellulose nanocrystals (CNC) and bacterial cellulose. The first two types can be produced from any cellulose source by employing different preparation method via chemical treatment (acid hydrolysis) and mechanical disintegration, respectively. Bacterial cellulose on the other hand is produced by bacteria of Gluconobacter genera such as Acetobacter xylinum.

Ever since the pioneering work on the isolation of NFCs from fibres numerous studies have been conducted to produce NFCs from various starting materials namely wood, non-wood and agricultural residues [14]. Integrating pulp mill with NFCs production could offer new opportunities for the pulp and paper industry. Thus, the reason Shorea roxburghii was selected in this study was due to its availability in Malaysia from plantation forest and also conversion of Shorea pulp into (NFCs) could create product of good economic value.

The previous research studies have shown the effectiveness of the use of nanocellulose filter paper. An example, pure nanocellulose filter paper by using Cladophora algae cellulose featuring an average pore size of 19 nm and specific surface area of 88 m^2 $g{-1}$. The predominant majority of pores is ≤ 30 nm. The nanocellulose heat pressing produced membranes with an average thickness of 70 μm and a total porosity of 35.00 %. For the first time a 100.00 % natural, unmodified nanofibrous polymer based membrane is demonstrated capable of removing virus particles solely based on the size exclusion principle, with log10 reduction value (LRV) ≥ 6.3, thereby matching the performance of industrial synthetic polymer virus removal filters [15]. Apart from the studies, research on the production of cellulose nanocrystals (CNC) cotton fiber nanocomposite filter papers have been successfully used as a simple and efficient bacterial filter removal performance. The convenient preparation, sustainable materials and low cost give this composite filter paper a potential application in water purification, especially in the developing countries. The CNC composite filter papers also show promising prospects and potential applications in many fields related to bacteria removal such as biomedicine, environmental protection, and food industry [16].

3. Methodology

This chapter discusses the list of materials, preparation and modification of the adsorbent, and the adsorption process. It is important in order to provide better understanding about the research process employed in this study. This chapter also describes the whole research process including the problem formulation process, the data interpretation process and dissemination of information. The formulation of research design helps to isolate and to enhance potential of contextual factor that are related to the topic of study. Appropriate research methodology will assist in achieving research objective by clearly showing the method of data acquisition, data analysis and to generate information.

3.1 Material and Method

Shorea roxburghii was obtained from a Forest Research Institute Malaysia (FRIM) Kepong, Selangor. Sulfuric acid (96.00 %) was purchased from Fisher Scientific (UK) while polyvinyl alcohol (PVA) (fully hydrolysed) was obtained from Sigma Aldrich (US). Sodium hydroxide, sodium sulfide, ethanol and toluene were purchased from R&M Chemicals (UK), glacial acetic acid from Ajax Chemicals (Australia) and sodium chlorite from Merck (USA).

3.2 Preparation of Adsorbents

3.2.1 Pulping and Bleaching

Kraft pulping for *Shorea roxburghii* was prepared by cooking wood chips in a rotary digester with 17.00 % active alkali (sodium hydroxide and sodium sulphide) at 170 °C for 3.5 hours with 1:3.5 of wood: liquor ratio. After that, the cooked pulp must be washed to remove the chemical substances contained in the pulp and to remove the mulky water by hydro pulper. In order to eliminate impurities, debris and uncooked fibres, the resulting pulp which has been cooked was screened with PTI Somerville fractionator. After the screening process, pulp was placed in the sack and spinning to reduce the water on the pulp. Then inserted the pulp into the Hobart Mixer, which separates the pulp. After pulping, the pulp was bleached via elemental chlorine free bleaching method using 5 - step bleaching sequences i.e. DEDED. The D refers to chlorine dioxide and E is extraction by sodium hydroxide. Based on table 1 showns the DEDED sequence for bleaching the wood pulps. Bleached pulp was used at this stage as a feedstock in the making of nanocellulose.

Table 1: The DEDED sequence for bleaching the wood pulps [17]

Chemical & Charge (%)	**D1**	**E1**	**D2**	**E2**	**D3**
Pulp Consistency (%)	ClO_2 3% Acetid Acid 3%	NaOH 1%	ClO_2 3% Acetid Acid 3%	NaOH 1%	ClO_2 3% Acetid Acid 3%
Time (Min)	120’ (2 Hour)	60’ (1 Hour)	90’ (1 Hour 30 min)	60’ (1 Hour)	90’ (1 Hour 30 min)
Temperature (°C)	70 °C	70 °C	70 °C	70 °C	70 °C

3.3 Preparation of Nanofibrils cellulose (NFCs)

Disintegrator, hand sheet, refining and grinding was used for preparation of Shorea roxburghii nanofibrils cellulose techniques for mechanical production of NFC. Bleach pulp was placed into container and half of water from the container was refilled. The time of disintegrator machine has been set and it will stop automatically after 30 minutes. The next process is to used hand sheet machines, with the sheet machine open, turn on the water and gently rub the surface of the wire with the fingers to clear away any adhering fibers. The machine was closed and filled it with half of water. The amount of stock needed was poured into the sheet machine. Water was added until the depth was about 350 mm above the surface of the forming wire. The sample was inserted into sheet former and waited for 5 seconds allowing entrapped air to leave and the fibres to settle.

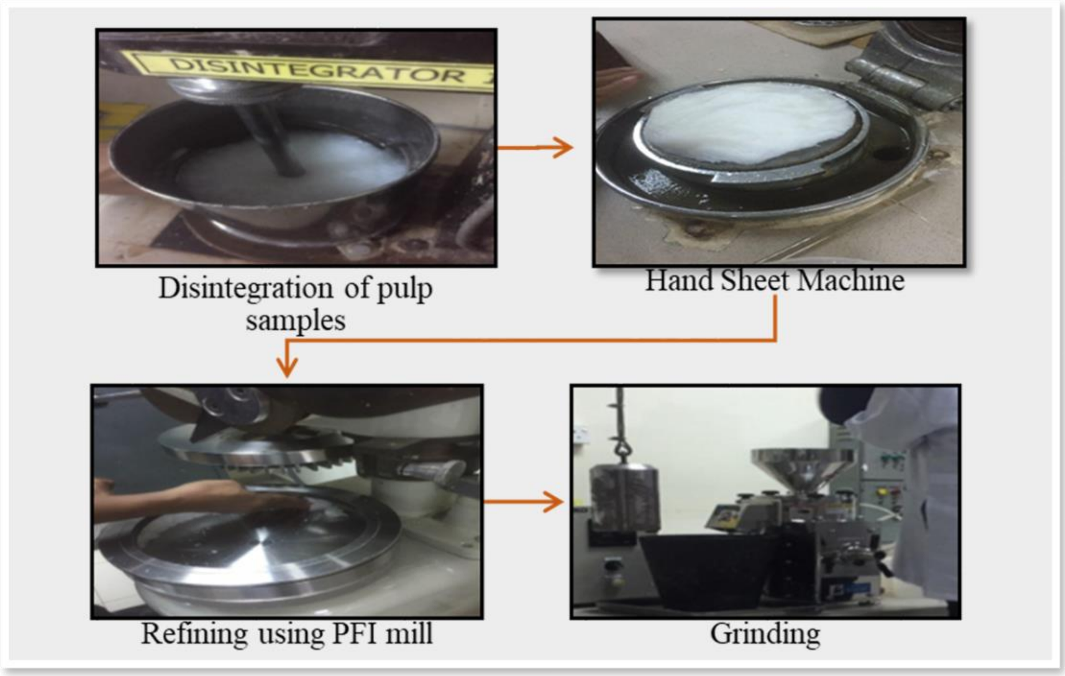

Figure 1: The process of Nanofibrils cellulose (NFCs) preparation

The refining by using PFI mill was performed by pasted the pulp of weighted 240 g around the bedplate, make sure the pulp is not too dry so that it sticks easily. The area was closed and switched on the PFI mill button. After 40000 second, switched off the button and removed the pulp sample from PFI mill bedplate and bar. Final process for preparation of Nanofibrils cellulose (NFCs) was grinding. The grinding machine was set to the specification. Both the pulp was put into the grinding machine which has to be processed 10 times. After 10 grinding times, take a small pulp to check the size of the nanoscale using a cellulose lab machine with a maximum size of 100 nm. If the size does not reach below 100 nm, the grinding process needs to do as much as 10 times again. The process of Nanofibrils cellulose (NFCs) preparation has been shown in the Figure 1

3.4 Manufacturing of nanocellulose filter paper

For making the nanocellulose filter paper, the thickness of the filter paper and the total volume of water to be combined with the pulp must be identified to find out the amount needed for the production of filter paper. It is based on the equation of 1 and 2

$$\text{Thickness (gsm)} = \frac{Weight\ (g)}{Area\ (cm^2)} \times 10\,000 \qquad \text{Eq. 1}$$

$$\text{Pulp consistency} = \frac{Oven\text{-}dry\ weight\ of\ pulp}{Weight\ of\ pulp + water} \times 100 \qquad \text{Eq. 2}$$

The consistency of Shorea roxburghii pulp was 1.22 g for 20 ml equivalent to 60 gsm for a piece of filter paper. The water through the porous structure was difficult for 100.00 % NFC filter paper, because the pores size of full NFC filter paper almost like a macroporous polymers pores size range of 50 nm to 1 µm [18]. For grade 1 qualitative Whatman filter paper – standard grades had 11 µm [19]. The filter paper had to be modified and the solution was 70.00 % of NFC and the bleaching pulp mixture was

30.0 %. The water can go through a pore filter by a drop from these modifications. Figure 2 shown the nanocellulose filter paper that was successfully produced.

Figure 2: Shorea roxburghii nanocellulose filter paper

3.5 Characterization of Shorea roxburghii nanocellulose filter paper.

3.5.1 FTIR

Fourier Transmission Infrared Spectroscopy i.e. FTIR spectra were collected on a Perkin Elmer Spectrum 100 spectrometer in absorbance mode using KBr sample discs. The spectra were recorded in the range of 450 to 4000 cm^{-1} [20].

3.5.2 Zeta Potential

Zeta potential determination was carried out using 0.10 % suspensions of NFC in deionized water using a Malvern Zetasizer ZS.

3.6 Wastewater Samples

The sample collection was carried out at the Anfi Textile Industry, Parit Sri Sulong, Johor. Wastewater effluent was collected by using grab sampling due to concerns over sample stability for stored composites it allows the analysis of unstable parameters such as pH, dissolved oxygen and temperature [21].

3.6.1 Wastewater Characteristic

In order to perform a physico-chemical characterization of the water samples, the American Public Health Association's Standard methods for the examination of water and wastewater were applied. In each of the samples, the following water quality parameters: pH, turbidity, initial concentration (ADMI), Biological Oxygen Demand (BOD), Dissolved Oxygen (DO) and Chemical Oxygen Demand (COD) were determined at the laboratory room.

3.7 Batch Adsorption Study

Adsorbent of NFC was produced to be nanocellulose filter paper. The dye effluent was prepared into measuring cylinders with volume 20 ml used for filtration and then dye effluent was filter using filtration pump. After filtration end, by using DR6000TM UV VIS Spectrophotometer (HACH), the initial and remaining dye was analyzed. This experiment was carried out at room temperature. For determining the uptake of the dye, all-inclusive sets of experiments were performed at different time intervals 5, 10, 20,30,40 and 60 minutes and pH is 4,6 and 8. A initial dye concentration (ADMI) from 100, 150, 200, 250 and 300 was also used to perform this experiment. The removal of percentage was calculated using equation:

$$\% \text{ removal} = \frac{co-ci}{co} \text{ x } 100\% \qquad \text{Eq. 3}$$

Where, C_0 for the liquid-phase concentrations of dye at initial (ADMI); C_i, for the liquid-phase concentrations of dye at final (ADMI); % removal represents the percentage colour removal.

4.0 Result and Discussion

4.1 Characterization

4.1.1 Chemical composition

Chemical composition of Meranti wood is summarized in Table 2. Meranti wood value of cellulose and lignin are in the range of Malaysian hardwoods i.e. 41.58 % for ά- cellulose and 33.56 % for lignin [22].

Table 2: Chemical composition of Meranti wood

Compositions	Content (%, w/w)
Cellulose (alpha)	41.58
Hemicellulose	32.81
Lignin	33.56
Extractives	3.08
Ash	0.64

4.1.2 Scanning electron microscopy (SEM)

SEM has a resolution between that of optical microscopy and that of TEM or AFM, and permits observation of MFC and NFC from the millimetre to true nanometre scale [23]. NFCs had nanofibrils with a uniform diameter of approximately 15 nm. [24]. Figure 3 shows SEM images of MFC (left panel) and NFC (right panel); the fibril structure differs significantly between MFC and NFC, as MFC has many large-scale structures (i.e., fibres) remaining after 30 mechanical treatments, while NFC has thinner fibrils.

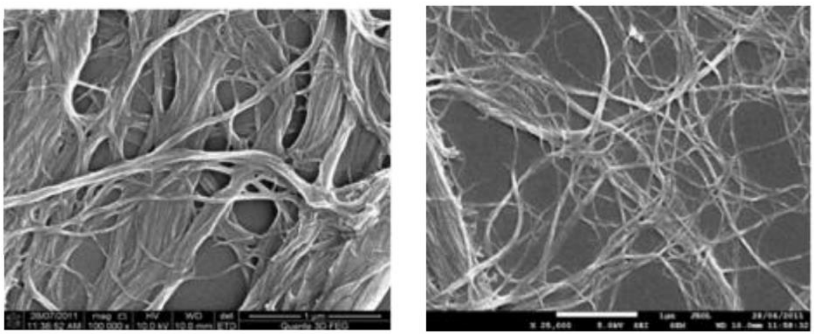

Figure 3: SEM images of MFC (left) and NFC (right) [25]

4.1.3 Modification of nanocellulose filter paper

Following the production of *shorea roxburghii* nanocellulose filter paper, the use of 100.0 % NFC was not possible as water could not penetrate the filter paper. The solution is to mix 70.0 % NFC and 30.0 % bleaching pulp. The result of the mixture found that water can penetrate the filter paper. This matter is because the cellulose fibrils were consisting of microfibrils with 3-4 nm in dimension and up to 20 nm for wood cell wall and for the short cellulose nanofibrils about 1~3 μm in length [26], even the membrane filter pore size 0.22 μm.

4.1.4 Nanocellulose filter paper

According to Voisin et al [27] studied indicate that freeze drying, followed by compaction of a mixture of sulfated CNCs and chitosan filter paper created microporous membranes with a thickness of 200 microns, capable of immobilizing positively-charged dye molecules. The pore diameters of the membranes were in the range of 10–13 nm, and the water flux was low (64 $Lm2 \cdot h-^{1} \cdot MPa-^{1}$). It is reported 84.0 %, 69.0 % and 98.0 % removal rates for Methyl Violet 2B, Rhodamine 6G, and Victoria Blue 2B from an aqueous solution at pH 5 when using freeze-dried affinity membranes functionalized with sulfated cellulose nanocrystals. Figure 4 show the visual appearance of freeze-dried membranes and SEM images showing the CNCs on the surface and bulk of the membrane. The membranes adsorbed 98.0 % of Victoria blue 2B from water.

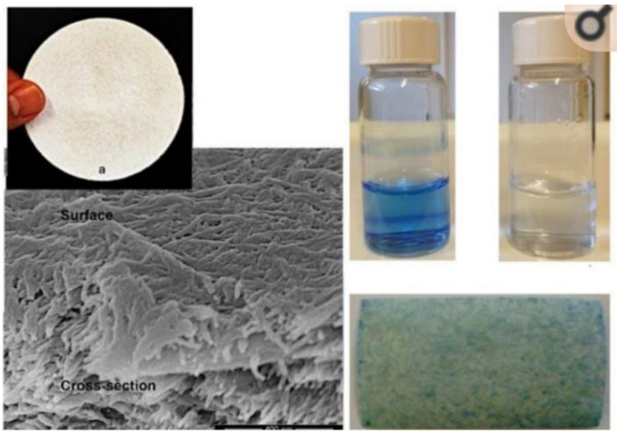

Figure 4: the visual appearance of freeze-dried membranes and SEM images showing the CNCs on the surface and bulk of the membrane. The membranes adsorbed 98.0 % of Victoria blue 2B from water

4.2 Wastewater characterization

The characteristics of composite wastewater at Anfi textile industries samples was based on the studied of Hamid et al (2020) [28]. The results obtained from the examination are shown in Table 3.

In the textile processing unit's pH is very much important factor. It is regulated at various steps for better results. The pH is also important in the dyeing step as the solubility of the dyes depends on it. The pH also changes with type of cloth processed. The result shows that the pH of textile wastewater was neutrally 7.5. Refer to National Environmental Quality Standards, the pH was set value 7.5 to 11.55 the effluents were alkaline in nature [29]. Thus the wastewater of textile industry is neutral to strong alkaline nature because in most of the steps caustic and other detergents of alkali nature are used in large quantity.

Biological Oxygen Demand (BOD_5) and Chemical Oxygen Demand (COD) of Anfi Industrial Textile wastewater was recorded between 31.12 mg/L and 602 mg/L. The BOD_5 reading of the wastewater sample higher than standard A, as well as the value of COD reading also very high comparing with standard A and B. Higher chemical oxygen demand of the wastewater is due to presence of process. higher chemical oxygen demand indicates the chemical pollution due to textile industry rather than biological pollution [28].

According to the result, it shows the textile wastewater samples's pH, DO, BOD_5, COD, turbidity and colour values was higher than the limit level set by National Water Quality Standard Malaysia. Therefore, continuing the treatment of textile wastewater is a necessary condition before it is disposed to the natural water system. Table 4 shown the parameter limits effluent of Standards A and Standard B.

Table 3: Characteristic of textile effluent wastewater

	pH	**Colour (ADMI)**	**COD (mg/L)**	**BOD (mg/L)**	**Turbidity (NTU)**	**Dissolved Oxygen (mg/L)**
Dye sample	7.5	548	602	31.12	39.43	5.96

Table 4 : Parameter limits of textile effluent Fifth Schedule (Environmental Quality (Industrial Effluents) [30]

Parameter	Standard A	Standard B
pH	6.0 – 9.0	5.5 – 9.0
Dissolved Oxygen (DO)	-	-
Total Suspended Solid (TSS)	50 mg/L	100 mg/L
Total Dissolved Solid (TDS)	-	-
Colour	100 ADMI	200 ADMI
Biological Oxygen Demand (BOD)	20 mg/L	50 mg/L
Chemical Oxygen Demand (COD)	80 mg/L	250 /L

5. Conclusion

In conclusion, nanofibrillated cellulose (NFC) filter paper was successfully produced from Shorea roxburghii bleached pulp, only the results and discussions from this study need to be done based on the results of other researchers, due to unavoidable issues that happened. Following the other studied, it shown that the nanocellulose filter paper can treated dye removal because pure nanocellulose filter paper by using Cladophora algae cellulose capable of removing virus particles and according to Karim et al studied indicate that mixture of sulfated CNCs and chitosan filter paper managed to remove 84.0 %, 69.0 % and 98.0 % removal rates for Methyl Violet 2B, Rhodamine 6G, and Victoria Blue 2B from an aqueous solution at pH 5, it is possible to design and optimize an economical treatment process for the dye removal from industrial effluents.. However, this study needs to be continued to get accurate results.

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