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Barrier Performance of Spray Coated Cellulose Nanofiber–Montmorillonite (MMT) Composites

Kirubanandan Shanmugam*

**Department of Biotechnology, Sree Sastha Institute of Engineering and Technology, Sree Sastha Nagar, Chembarabakkam, Chennai – 600123, Tamilnadu, India.*

*Corresponding Email: *Kirubanandan.shanmugam@gmail.com*

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Abstract:** Cellulose nanofibers are one of the most promising nanomaterials for developing high-performance paper-based packaging. This nanomaterial has renewable, recyclable, biodegradable and eco-friendly substrates in nature. While cellulose nanofiber layers have very lowest oxygen permeability when comparing with synthetic plastics, their water vapour permeability is significantly higher than that of conventional packaging plastics, such as Low-Density Poly-ethylene (LDPE). Water vapour permeability has been decreased by forming composites of cellulose nanofibers and inorganic nanoparticles, such as Montmorillonite (MMT) clay. However, the addition of the nanoparticles further reduces the already poor drainage when layers are formed through vacuum filtration. The method for spray-coating a cellulose nanofiber-montmorillonite layer is developed to produce composite. It improves both the ease of preparation and reduces the water vapour permeability. The effect of high-pressure homogenization treatment to the suspension before composite preparation on the barrier performance is also investigated. The permeability could be reduced below that achieved with vacuum filtration by adding up to 20 wt. % Montmorillonite and dispersing Montmorillonite with two passes in a high-pressure homogeniser. With Montmorillonite addition above 20 wt. %, the water vapour permeability started to increase due to aggregation of the Montmorillonite. At the optimal addition level, the best performance achieved with spraying was a water vapour permeability of 8.3×10^{-12} g/m.s.pa. The air permeability of composite is evaluated to be less than 0.003 µm/Pa.s. This value confirms an impermeable composite for packaging applications. Considering the barrier performance, spray coated nanocomposites can perform as an effective barrier material and a potential alternative to synthetic plastics.

***Keywords:** Cellulose nanofibre, MMT, Nanocomposite, Spraying, Vacuum Filtration, Coating, Water Vapour Permeability, Air Permeability and Barrier Materials.*

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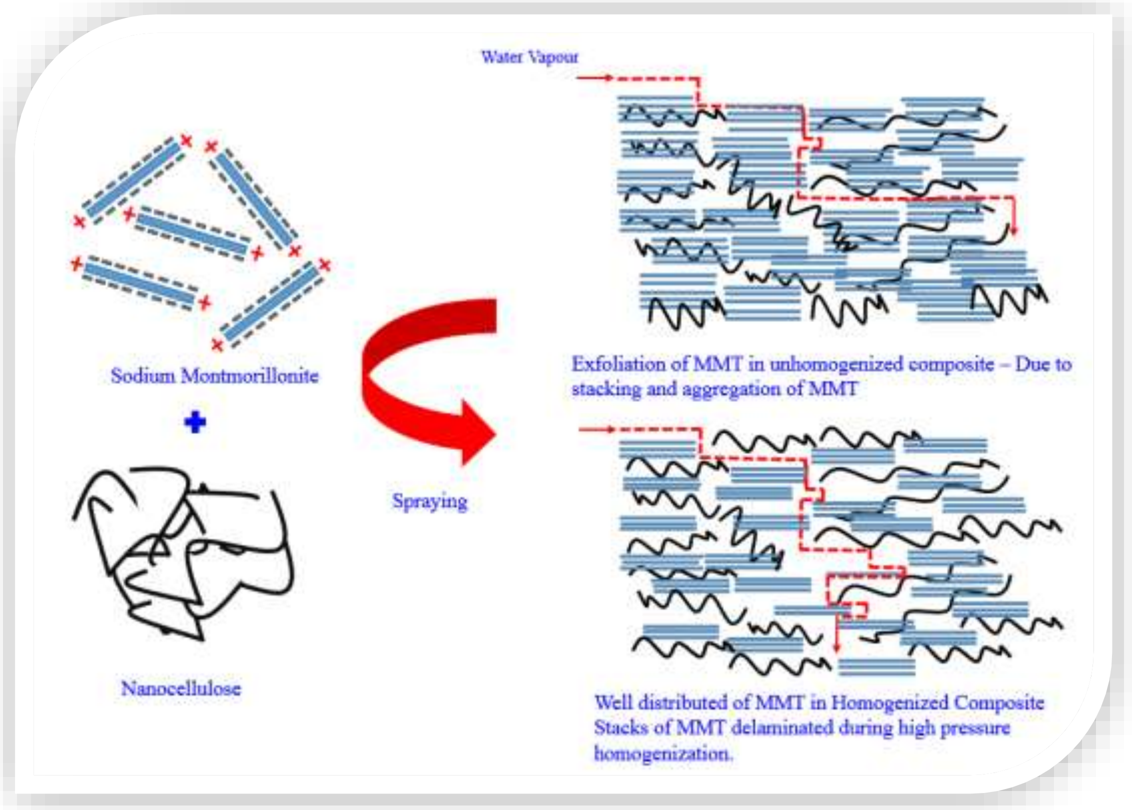
Graphical Abstract



1. INTRODUCTION

Synthetic Plastics or Polymers from the petroleum sources are used as the predominant packaging materials. However, these materials are not biodegradable and cause an environmental threat to the earth via land pollution with synthetic plastic dump. Synthetic plastics are one of the sources for forming microplastics and nano plastics contaminating the environment and also penetrating into food chain [Muncke, J. 2021]. This is why, Biopolymers are considered for the development of food packaging materials and also alternative to replace synthetic plastics in this application [Nair, S.S., et el 2014].
Cellulose is the predominant biopolymer with fibrous structure used for the packaging application in the form of papers and paper boards. Paper and paper boards are cellulose macrofibre substrates having wide pores in the substrates. These wide pores allows considerable amount of water vapour and air across the paper and paper board. It results in poor barrier performance of the paper and paper board. It was resolved by the coating of paper substrates with synthetic plastics, wax and extrusion with aluminium sheet. However, this approach is not an eco-friendly and not recyclable [Nair, S.S., et el 2014] [Shanmugam, K, 2021].

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The coating of Cellulose nanofibers on the paper substrates is a sustainable approach and achieving the circular economy requirements [Shanmugam, K, 2021]. Cellulose nanofiber is a sustainable fibre based nanomaterials produced from wood pulp via mechanical disintegration, chemical process and enzymatic process. The diameter of cellulose nanofiber varies from 5 to 70 nm with length of 8 microns [Souza, E, 2021] The spraying CNF on the paper substrates firstly fill the surface pores in the paper and forms a barrier layer on the paper substrates. These layers acts as barrier protection of the CNF coated paper substrates against water vapour, oxygen and air and increase the barrier performances [Shanmugam, K, 2021]. CNF is a biodegradable nanomaterial and potential for recyclability and providing circular economy [Ahankari, S.S et al, 2021].

Recently, self-standing CNF film is a good barrier material for replacing the synthetic plastics in food packaging application [Shanmugam, K, 2021] [Shanmugam, K,2017]. CNF film was recently prepared via the spraying process. In this process, the CNF suspension was sprayed on the polished metal surface such as polished stainless-steel plate and then dried into a standard laboratory condition. The process yields free standing CNF film easily peeled from the stainless-steel plate after drying process [Shanmugam, K, 2021]. The spray coated CNF film was used as barrier material, membrane and substrates for flexible electronics [Shanmugam, K,2019]. The main application for spray coated CNF film was barrier application or food packaging application [Shanmugam, K., 2020]. However, pure CNF film has some limitations such as poor water vapour permeability at high relative humidity and weakening cellulose nanofibrils at high moisture surroundings. To resolve this limitation, the incorporation of nano-montmorillonite (Nano-MMT) into the cellulose nanofiber matrix results in elevation of water vapour permeability and mechanical properties. Nano-MMT impregnated into the cellulose nanofibre matrix increase the tortuous pathway for transfer of water vapour and oxygen. As a result, the barrier performance of CNF-MMT composite was enhanced [Shanmugam, K., 2021].

This paper investigates the preparation of CNF-MMT composite via spraying CNF-MMT suspension on the polished metal plate [Shanmugam, K., 2021] and then evaluated the water vapour permeability of the composites. In addition to that, the barrier mechanism of the CNF-MMT composite has been explained.

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Background

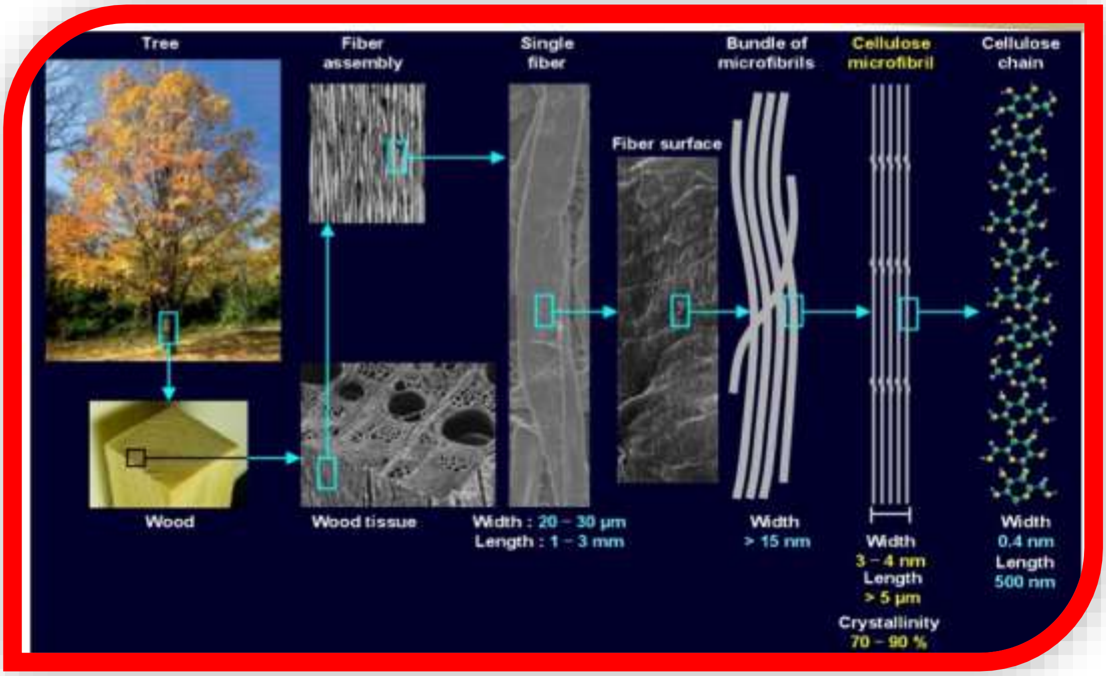

Figure 01 – Cellulose Nanofibre/Nanocellulose from Wood

Figure 1 shows the cellulose nanofiber produced from the wood pulps via the mechanical disintegration of cellulose pulps, chemical methods such as acid hydrolysis and enzymatic process [Shanmugam, K., 2021]. Figure 1 shows the hierarchy of cellulose nanofibers from wood. Cellulose is the main component of the wood and defibrillated into cellulose microfibres and cellulose nanofibre. The diameter of cellulose nanofibrils vary from 5 nm to 100 nm and depends on the type of processing. Normally the cellulose nanofibre consists of two major regions namely such as amorphous and crystalline regions [Ahankari, S.S., 2021]. These regions produce the tortuous pathway for transfer of gaseous substances such as water vapour, oxygen and air. This phenomenon increased the barrier potential of cellulose nanofibre [Azeredo, H.M., 2017]. The length of cellulose nanofibrils in the CNF normally greater than 5 microns. Due to the nano size of CNF, it has considerable properties for fabrication of various functional materials. CNF has high mechanical strength and high E-Modulus for the development of packaging [Syverud, K., 2009] and membranes [Shanmugam K, 2021]. The CNF has a good barrier against Oxygen and water vapour [Syverud, K., 2009]. So it can be potential alternative for synthetic plastic packaging.

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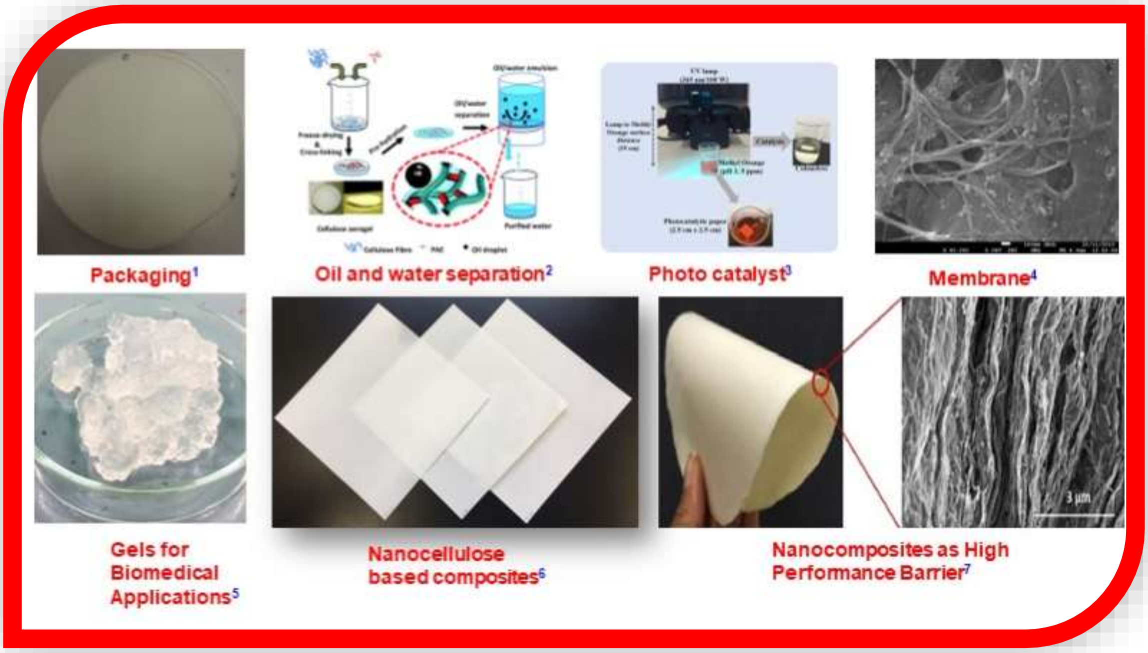

Figure 2 – Functional Application of Nanocellulose

Figure 2 reveals the functional application of nanocellulose/cellulose nanofibre. The CNF has good barrier against water vapour and oxygen [Syverud, K., 2009] . So it can be used as packaging material and coating CNF on the paper substrates for enhancing barrier performance of the paper substrates [Shanmugam K, 2021]. The CNF film was used as a membrane for Oil and water separation and waste water treatment. The CNF film can be used as a base substrate for incorporating photo catalyst such as titanium di oxide for the photo catalyst membrane. This can be useful for treatment of wastewater treatment containing the substances susceptible to photochemical reaction. The CNF gels was used as a base biomaterial for diagnostic and therapeutic application. The inorganics was incorporated into the CNF suspension to make nanocomposites which used as barrier materials and food packaging applications [Shanmugam K, 2021].

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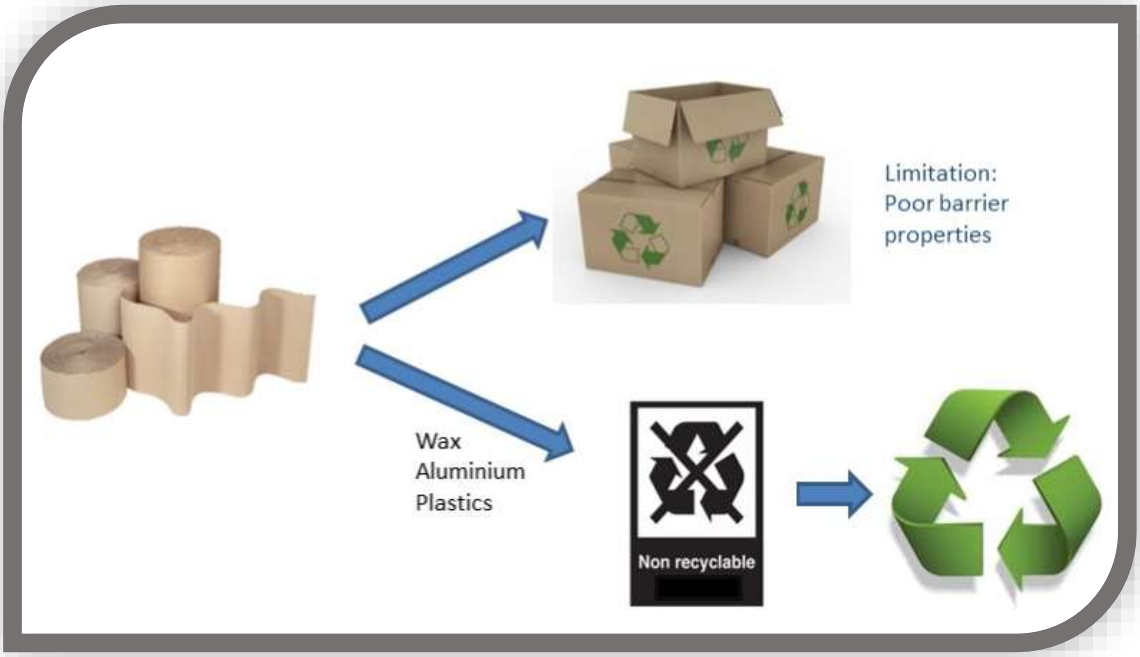

Figure 3 – Coating of Nanocellulose on the paper substrates

Figure 3 shows the necessary for coating CNF on the paper substrates. Normally, Paper substrates was used as one of the conventional packaging wraps for various packaging applications. However, paper substrates have wide pores for allowing considerable amount of water vapour and oxygen. It results in poor barrier performance and it can be resolved by the coating paper with wax, aluminium and synthetic plastics. These coating materials are not biodegradable, not recyclable and a threat to environment. To overcome these issues, CNF coating on the paper substrates is a novel approach and CNF fill the surface pores and forms barrier layers on the paper substrates. The coating on the paper substrates was performed via the spraying CNF suspension on the paper. This triggers the ideation for formation of free standing CNF film through spray coating [Shanmugam K, 2021].

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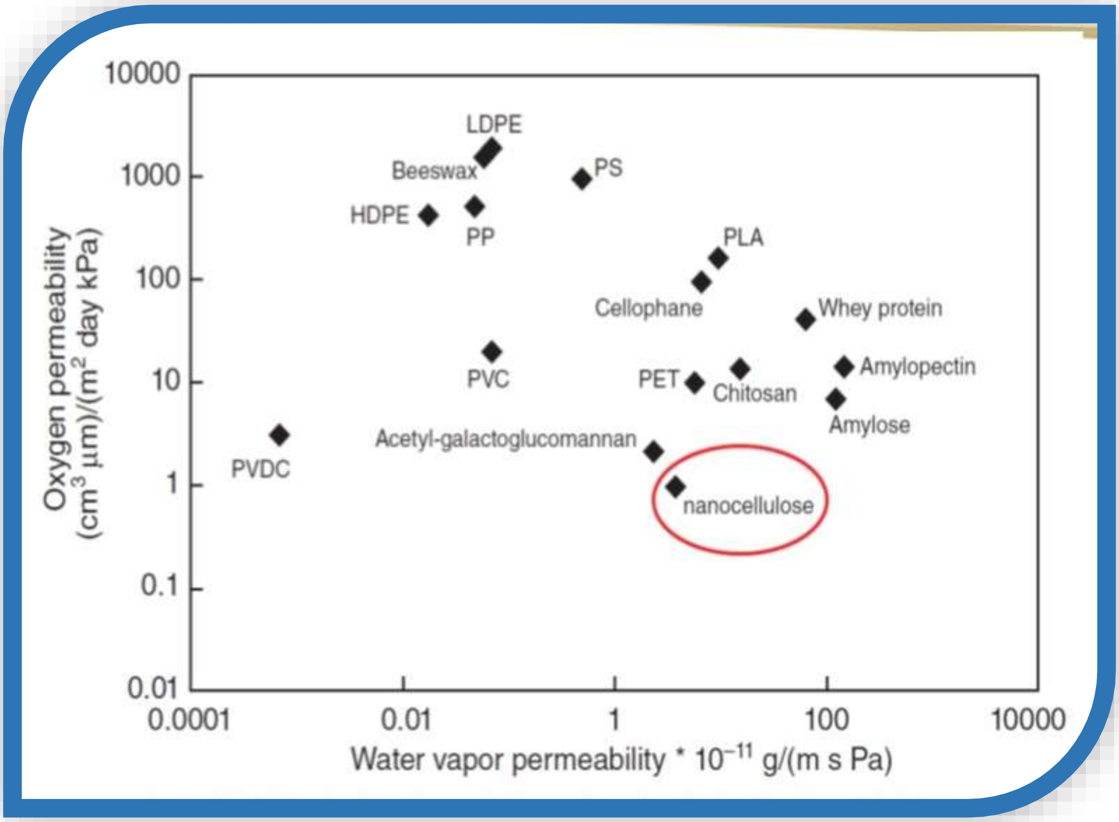

Figure 4 – Barrier Potential of Nanocellulose/Cellulose Nanofibre.

Figure 4 reveals the plot between water vapour permeability and oxygen permeability of the biopolymer and synthetic polymers. The above plot confirms that CNF has good barrier against oxygen and showed better barrier performance than that of other biopolymers, biodegradable and synthetic plastics. However, CNF/NC has poor barrier against the water vapour and high water vapour permeability comparing with synthetic plastics and low water vapour permeability comparing with other biopolymers. In packaging fields, synthetic plastics are the predominant play than that of other polymers. This is why, the WVP improvement of NC/CNF is required and to bring WVP value nearing the synthetic plastics. This was attempted by the incorporation of nanoclay into CNF suspension to form the composite which has lower WVP than that of Pure CNF film. Figure 5 shows the schematic diagram of Pure CNF film. CNF consists of two regions such as amorphous and crystalline regions. These crystalline regions produce the tortuous pathway for transfer of water vapour [Shanmugam K, 2021] [Bras et al 2017].

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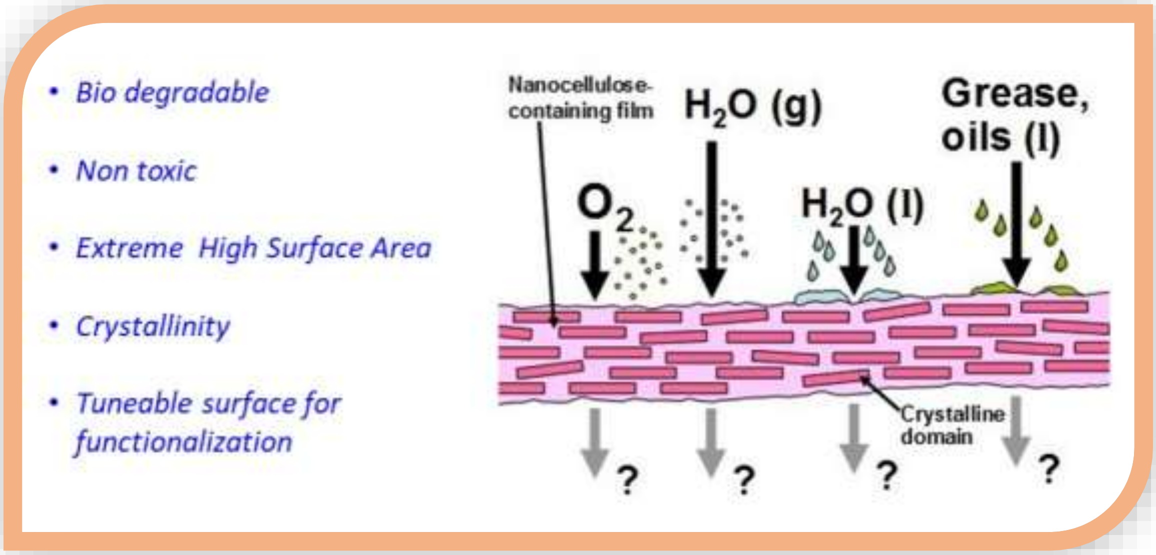

Figure 5 – Barrier Mechanism of Nanocellulose

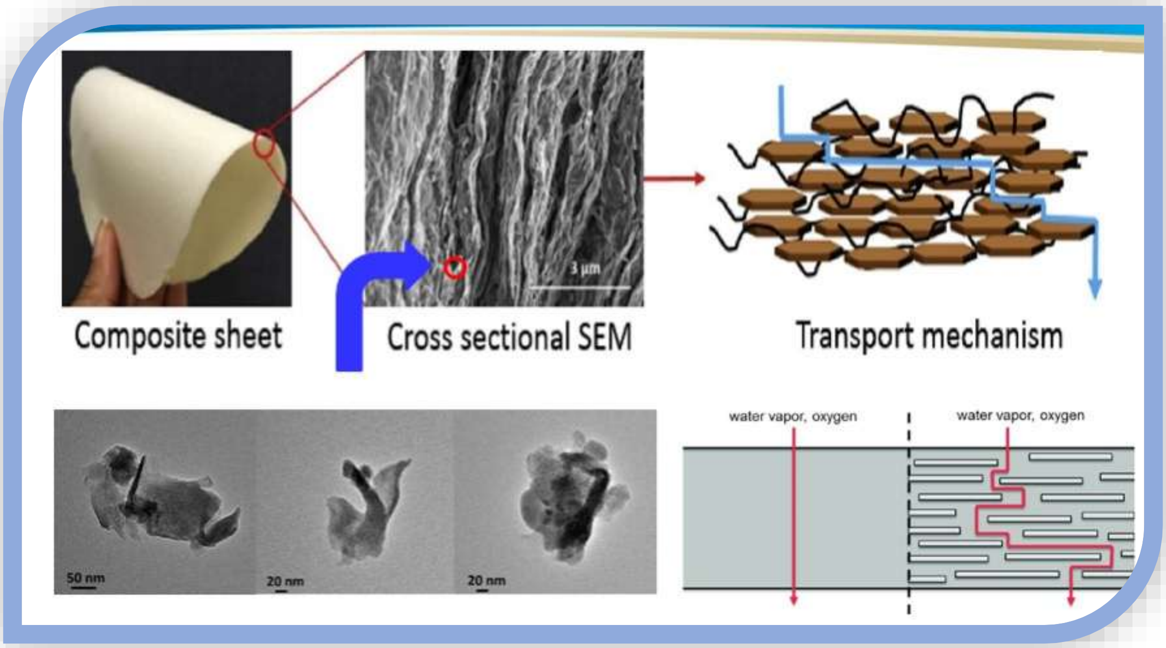

Figure 6; Incorporation of Nanoclay in Cellulose nanofibre

Figure 6 shows the concept of incorporation of nanoclay into cellulose nanofibers matrix. The above figure shows various shape of MMT incorporated into cellulose nanofibers matrix. The resulting composites have complex tortuous pathway for water vapour transfer than that of pure CNF film. This is why the CNF -MMT was developed via spraying. Apart from the barrier

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performance of CNF-MMT composite, the mechanical properties of the composite were also promoted [Shanmugam K, 2021].

This paper deals the following research investigation for the development of CNF-MMT composites,

(1). Spraying CNF -MMT suspension on the polished metal plates to fabricate the CNF – MMT composite

(2) Perform high pressure homogenization of CNF-MMT suspension and spraying on the polished plates

(3) Evaluating WVP of the spray coated composites

2. MATERIALS AND METHODS

Cellulose nanofibre also called as nanocellulose, microfibriallated cellulose, nanofibrillated cellulose, Cellulose nanofibre, etc. In this manuscript, Cellulose nanofibre or Nanocellulose consistently mentioned. The spraying of nanocellulose suspension was carried out on the surface of polished circular stainless-steel plate. The base surface of the polished stainless-steel plate was very smooth and very minimal scratches. Figure 07 shows the experimental setup for the proof of concept for spraying nanocellulose on the stainless-steel plate. In this experimental set up, there are professional spray system, conveyor and seating arrangement for keeping the stainless-steel plate after sprayed nanocellulose suspension. Nanocellulose (NC) supplied from DAICEL Chemical Industries Limited (Celish KY-100S) was utilised CNF suspension used to prepare free standing films via spraying. The suspension consistencies of CNF suspension ranges from 0.5 to 2.0 wt. %, prepared by diluting the solid content (CNF) of 25 wt. % with distilled water and mixing for 15,000 revolutions in a disintegrator. The viscosity of the CNF suspension was estimated by the flow cup method. In this method, the process of coating fluid flow through an orifice was used as a relative measurement of kinematic viscosity, with the results expressed in seconds of flow time in DIN-Seconds. In this case of fabrication of CNF-MMT composite, the desired quantity of MMT was added into the CNF suspension and fibrillated via high pressure homogenization in a high pressure GEA homogenizer. The two pass of the suspension of CNF and MMT in the homogenizer was performed to achive the cellulose nanofibrils diameter of ̰20 nm. The reduction of raw cellulose nanofibrils from 73 nm to highly homogenized nanofibres 20 nm was enhanced the intercalation of MMT in the cellulose fibres matrix. The highly homoginized suspension of CNF and MMT was sprayed on the stainless steel plate to fabricate the nanocomposite. The percentage of MMT was varied from 0 wt.% to 75 wt.% in the 2.0 wt.% CNF suspension for the fabrication of composites. The wet spray coated suspension of Pure CNF and CNF-MMT sheet was dried under the standard laboratory conditions. The dried CNF film and composites were subjected to various characterization such as topography studies and barrier studies of the composites [Shanmugam K, 2021].

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Figure 7 – An experimental set up for spraying CNF suspension on the stainless steel plate

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Figure 8 – An experimental set up for spraying CNF -MMT suspension on the stainless steel plate.

Figure 8 shows the sequences and steps involved in the fabrication of raw CNF-MMT composites and composites fabricated after high pressure homogenization of CNF-MMT in a high pressure homogenizer.

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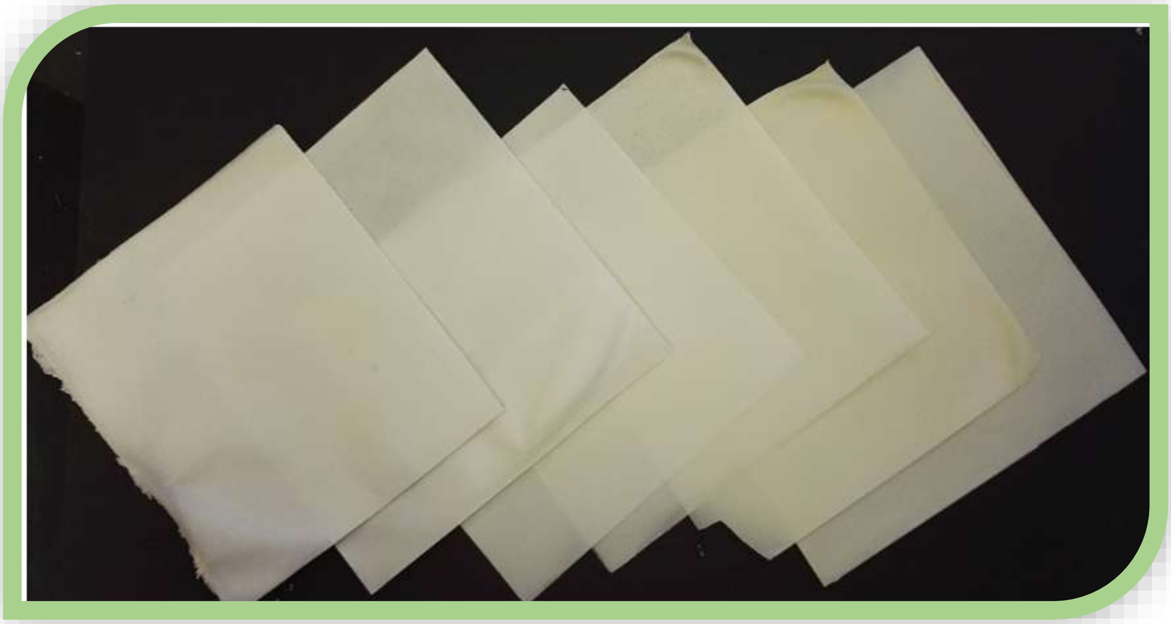

Figure 9 – Spray Coated CNF-MMT composite

3. RESULT AND DISCUSSION

The spraying CNF-MMT on the polished metal plates produced wet form of the nanocomposite. The coated wet composite was dried in a standard laboratory condition. The dried composite was peeled from the stainless steel plate and subjected to various characterization. The coated nanocomposite has two unique surface namely rough and smooth surfaces [Shanmugam K, 2021]. The rough surface of the composite has porous and very high roughness where as the smooth surface has shiny and glossy and less roughness. The smoothness of the surface was replicated from the stainless-steel plate [[Shanmugam K, 2021]. This smoothness has been used for developing various functional materials such as substrate for flexible electronics, solar cell and electrodes etc,. The MMT content varied in the CNF suspension gives as additional quality to the composite via yellowing of the composite. The surface topography and morphology of the composite was evaluated by the scanning electron microscopy. Further, the cross-sectional investigation of the composite was also investigated to study the intercalation of MMT in cellulose nanofibrils using scanning electron microscopy. Figure 9 reveals the spray coated CNF – MMT composite where the yellowing on the surface composite was increased with MMT content.

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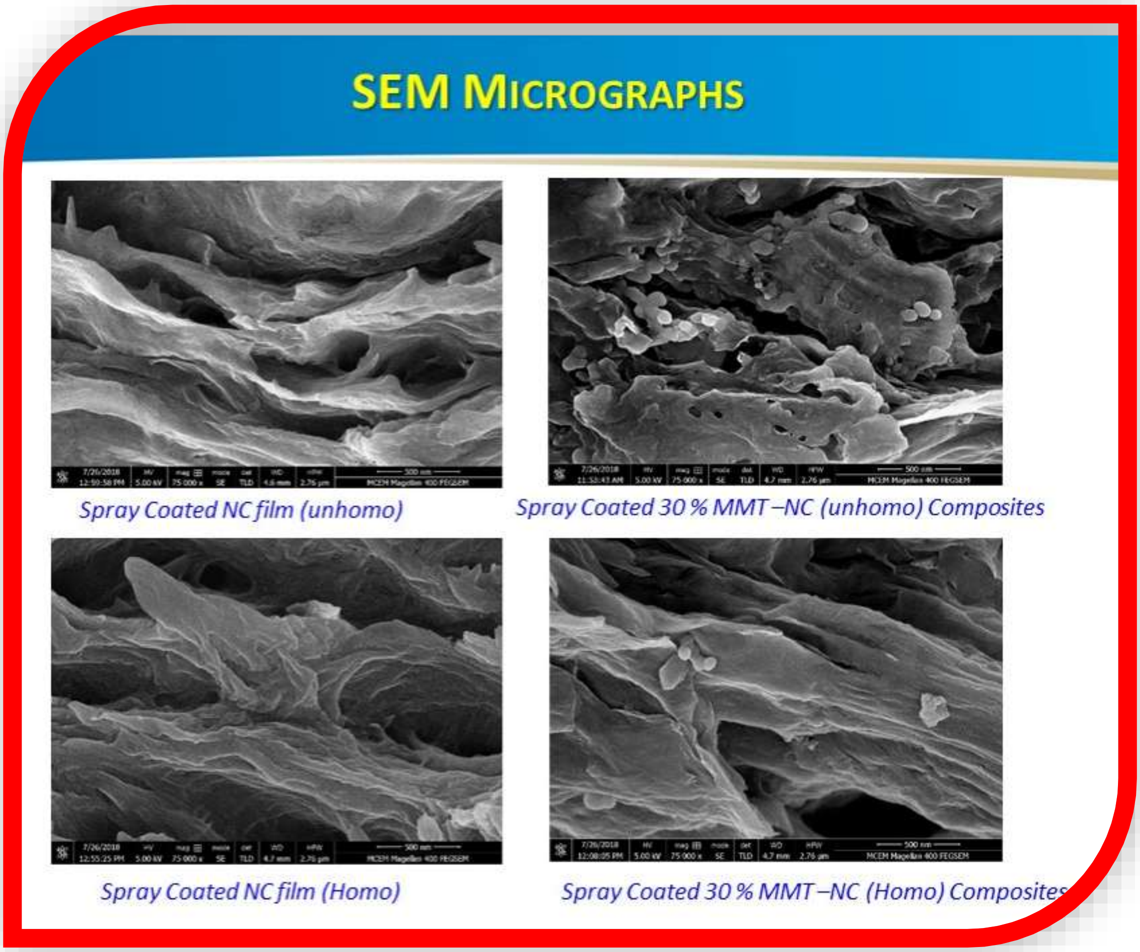

Figure 10 – SEM micrographs of Pure CNF film and 30 wt% MMT-CNF composite

Figure 10 reveals the morphology and topography of the pure CNF film and CNF – MMT composite. These are cross sectional images of the film and composite. In pure CNF film , the complex tortuous pathway was created by cellulose nanofibrils. In the case of composites, the MMT particle was intercalated in the cellulose nanofibrils network. High pressure homogenization of CNF-MMT produce the CNF supension well mixed with MMT resulting high performance composite for packaging applications. In Raw CNF- MMT composite, MMT particles was aggregated to decrease the barrier potential. The SEM image (Spary Coated 30 wt.
% MMT – NC (Unhomo) composites shows the MMT particles was aggregated. This aggrgation was uable to produce longest tortuous pathway for transfer of water vapour. In the case of homogenied CNF -MMT composite
Figure 11 - SEM micrographs of surface of the CNF film and composite.

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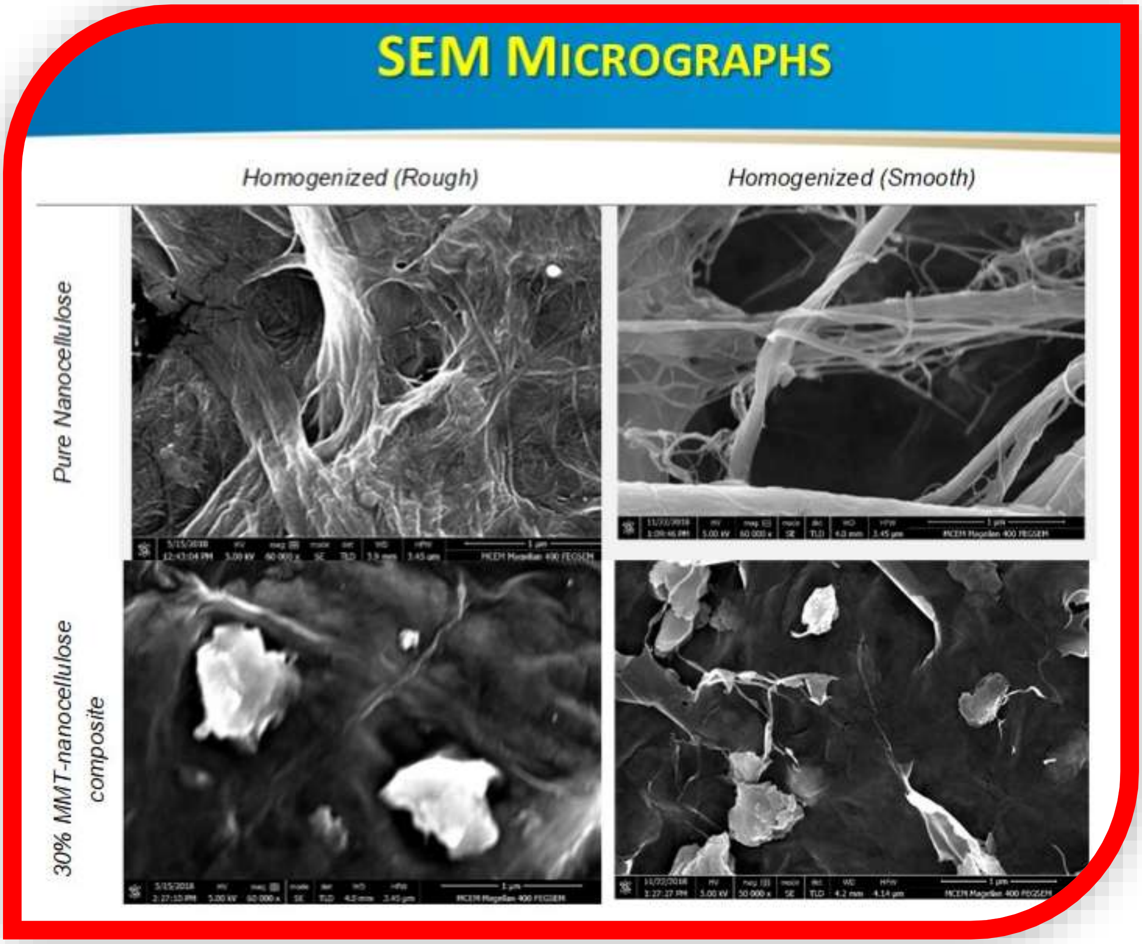

Figure 11 reveals the surface of CNF film and their composites via SEM micrographs. The MMT was scattered on the surface of the CNF -MMT composite. The spraying operation gives the composite with MMT distribution. During the high-pressure homogenization of CNF – MMT suspension, the cellulose nanofibrils and MMT are well mixed and MMT was distributed in the cellulose nanofibrils network during the mixing in high pressure homogenization process.

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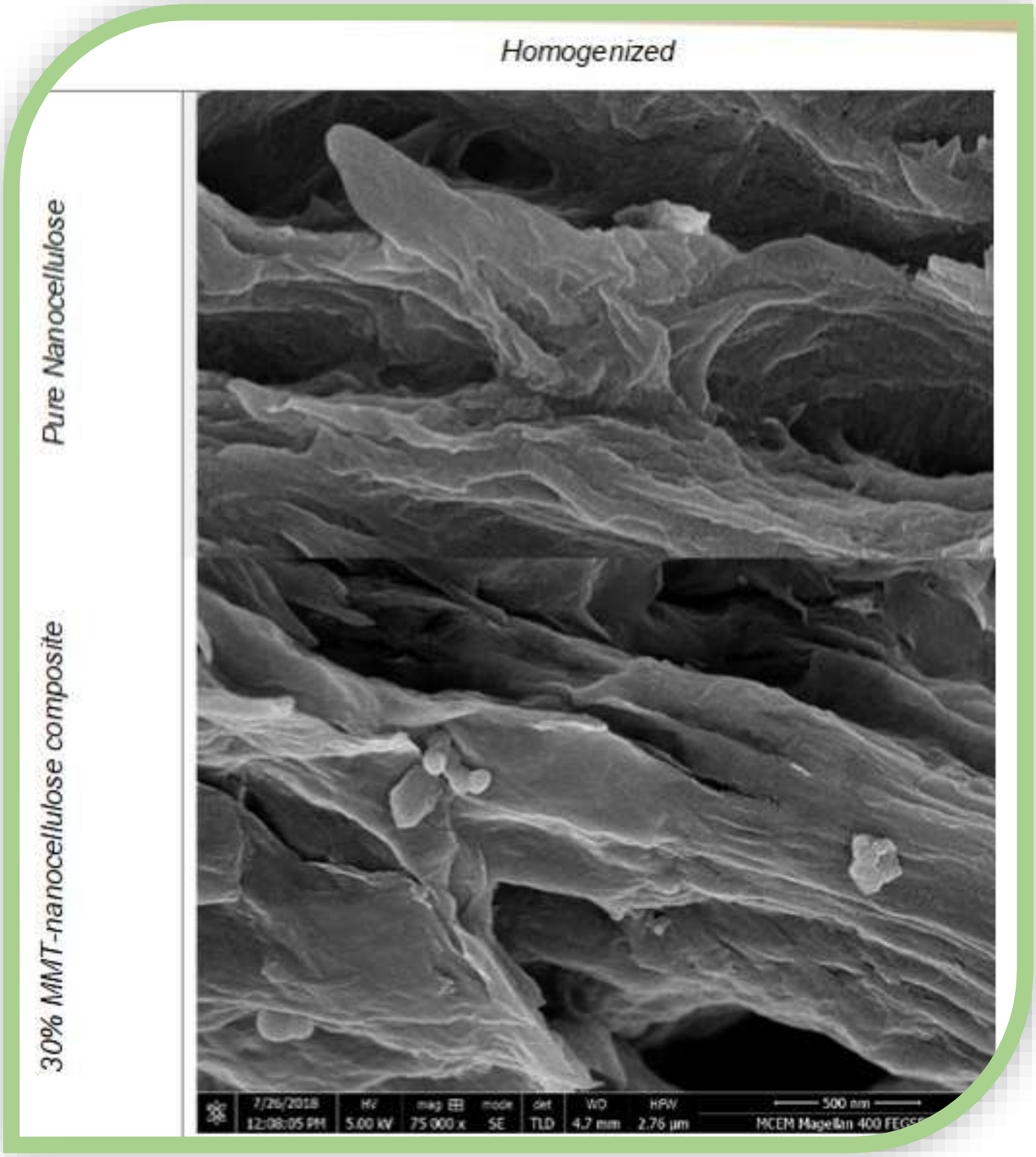

Figure 12 - SEM micrograph of Homogenised CNF – MMT composite

Figure 12 shows the SEM micrograph of the cross-sectional of CNF – MMT composite. Homogenization provides the well distribution of MMT in cellulose nanofibrils and this was seen in the cross section of the composite.

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Figure 13 – Air Permeance of CNF-MMT Composite

Figure 13 shows the air permeance value of the spray coated composite. The air permeance of the composite was evaluated to be less than 0.003 µm/Pa.S which is the maximum limit of measuring capacity of the testing instrument. This value confirms that the composite was highly impermeable against air and a potential barrier material against air and oxygen. The percentage of MMT in composite is independent of air permeance value. Spraying CNF-MMT suspension on the polished stainless steel plate was able to produce a compact composite which is impermeable against air.

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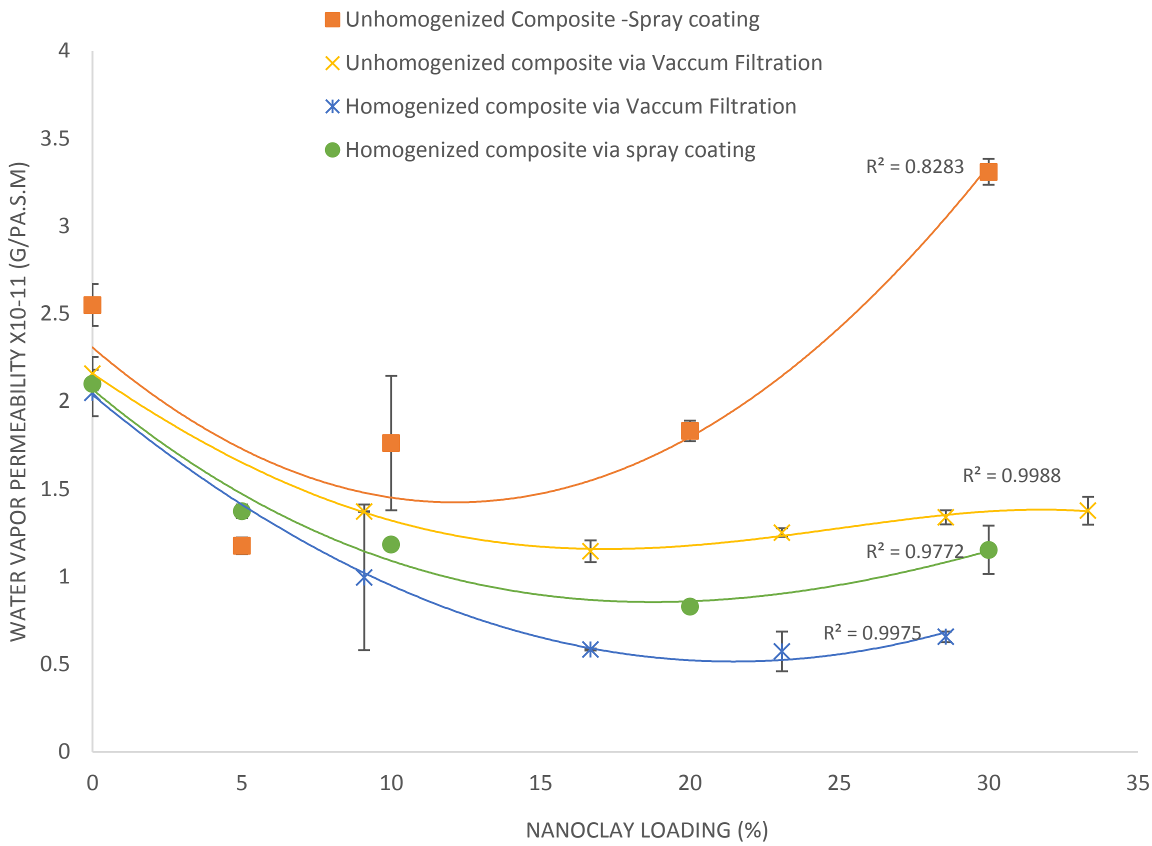

Figure 14 - Water vapour permeability of the spray coated CNF-MMT composite and compared with composite prepared via vacuum filtration

Figure 14 reveals the effect of MMT addition on the CNF suspension for the development of high-performance composite against water vapour transport. The plot 14 reveals the composite from homogenization has good barrier against water vapour than that of composite prepared from raw cellulose nanofiber. This is the case for both types of fabrication methods such as vacuum filtration and spray coating. In all case, the composite's WVP was reduced from 2-2.5 $x10^{-11}$ (g/Pa.s.m) to 1- 1.5 $x10^{-11}$ (g/Pa.s.m) with the addition of 10 wt.% MMT in 2 wt.% CNF. Beyond 10 wt.% MMT addition, the WVP was started to increase with MMT addition. This was happened due to the aggregation of MMT particles in cellulose nanofibrils network and additionally bentonite was highly hygroscopic nanoclay and absorbs a considerable quantity of water vapour in the composites. Up to 10 wt.% of MMT addition in the composite, the WVP of the composite was reduced and comparable with synthetic plastics. High pressure homogenization of CNF -MMT suspension reduced the diameter of cellulose fibrils and allowed to intercalate MMT nanoclay particles in cellulose nanofibrils network. The resulting composite via either spray coating or vacuum filtration has high performance barrier against water vapour permeance [Shanmugam K, 2021].
Vacuum filtration is the most common technique for fabrication of free-standing cellulose nanofibre films and their composite. However, the drainage time in filtration process was

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exponentially increased with the CNF suspension consistency and MMT content in the formulation. Normally, 3 or 4 hours to 10 min were required to form the free-standing film or composite. However, this time achieved only for dilute CNF suspension and the draining time will be increased with solid concentration/fibre concentration. Spraying is the fastest process in the formation of free-standing film and their composite. The operation time of the spraying has independent of suspension consistency and MMT content in the CNF suspension. The operation time for forming 15.9 cm diameter free standing CNF film or composite was less than 1 minute. When comparing with vacuum filtration, spraying has potential for scale up for large scale production of composites [Shanmugam K, 2021].

Barrier Polymers	Water Transmission Rate (g/m^2 day)	Average Thickness of the film (μm)	Water Vapour Permeability (g/m.s.Pa) x 10^{-11}
Spray Coated Composite (20 wt.% MMT)	12.5±0.4	80.2±1.8	0.83
Vacuum Filtered composite (23 wt.% MMT)	10.1	63.7	0.57
Polyethylene (PE)	16.8	18.3	0.1
Plasticized (PVC)	118.6	12.7	0.49
Aluminium Foil	2.4	18.3	0.014
LDPE	18	25	0.088
HDPE	9	25	0.044
PS	109 to 155	25	0.53 - 0.75
PP	6	25	0.029

Table 1 - Comparison of WVP of the spray coated composites with synthetic plastics

The Table 1 summarizes the WVP of the spray coated composites and synthetic plastics. This results confirming that the spray coated composite has comparable WVP with synthetic plastics.

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4. CONCLUSION

Cellulose nanofibre (CNF) - Montmorillonite (MMT) composite is a potential alternative barrier materials for synthetic packaging. The fabrication of CNF-MMT composite requires a fast process for scale up and large-scale production. Spraying is a rapid process for production of free-standing CNF film and their composites. Spraying CNF-MMT suspension on the stainless-steel plate produces unique surfaces of the composite namely rough surface and smooth surface. The spray coated CNF-MMT composite is an impermeable against air and good barrier against water vapour, confirming that the composite is a potential alternative for synthetic plastics. Further, the fibrillation of CNF - MMT via high pressure homogenization improves the water vapour barrier of the composite via spraying. Given this correspondence, the spraying CNF-MMT suspension produces a high-performance nanocomposite as barrier material against air and water vapour.

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