Journal of Cleaner Production 215 (2019) 226–231

Contents lists available at ScienceDirect

Journal of Cleaner Production

journal homepage: www.elsevier.com/locate/jclepro

The use of cellulose nanofibres to reduce the wet strength polymer quantity for development of cleaner filters

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ARTICLE INFO

Article history:
Received 19 October 2018
Received in revised form
10 December 2018
Accepted 2 January 2019
Available online 3 January 2019

Keywords:
Cellulose nanofibre
Wet strength
Composite
Filter
Adsorption

ABSTRACT

Polyamideamine-epichlorohydrin resin (PAE) is one of the most commonly used wet strength agents in the papermaking industry. However, adsorbable organic halogens (AOX) are known to be a toxic side product of the PAE manufacturing process; therefore, the use of PAE is restricted by regulation for food and medical applications. In this study, we investigated partially replacing the indirect food additive, PAE, with a renewable, biodegradable material, cellulose nanofibres (CNF), in order to drastically reduce the amount of PAE used while maintaining the same wet tensile strength. The concept is to replace covalent bonds by hydrogen bonds and to drastically increase bonding area. Depth-type filters were prepared with cellulose (30%), perlite (70%) and lesser amounts of PAE via papermaking technique. A small fraction of cellulose fibre composition was substituted with CNF while the PAE amount was gradually decreased. The substitution of positively charged PAE for negatively charged cellulose nanofibres switched the overall charge of the system from cationic to anionic. Therefore, two cationic polyelectrolytes, CPAM or PEI, were investigated to control the overall charge and adsorption performance of the filter system.

The substitution of CNF enabled PAE dosage to be reduced by over 95% while retaining the wet strength properties of the filters. The wet strength obtained from the small quantity of wet strength polymer could be further improved by increasing the curing temperature to 150 °C with a much shorter curing period. The filters with reduced PAE dosage have also improved adsorption of positively charged contaminants. However, for negatively charged contaminants a very minor amount (around 20 mg/g) of cationic polymer addition would be required to maintain the performance. Our study shows that partial replacement of conventional papermaking fibres with cellulose nanofibres allows us to reduce the quantity of wet strength polymer remarkably and achieve a sustainable and environmentally-friendly concept for filter manufacturing or for any paper product requiring wet strength.

1. Introduction

Wet strength is a critical property of certain paper products; it measures the ability of fibre network to maintain its strength when it is wetted with an aqueous solution. Wet strength is critical for hygiene products such as paper towel and tissue paper (Zakaria, 2004; Bajpai, 2018). Moreover, cellulosic filters that are used in liquid filtration also require a substantial wet strength to remain robust during operations.

Polyamideamine-epichlorohydrin (PAE) is by far the most common additive to manufacture wet strengthened papers (Obokata et al., 2005). Wet strength is developed through cross-linking the azetidinium groups with each other and with the cellulose (Obokata and Isogai, 2007). These reactions generate a partial water-proof barrier around the fibre to fibre contact point which restricts water from hydrolysing the hydrogen bonds when the sheets are re-wetted.

However, epichlorohydrin-based products, such as PAE, have significant drawbacks. Both the manufacture and use of epichlorohydrin can produce a high adsorbable organic halogen (AOX) content in the process wastewater (Hagiopol and Johnston, 2011). The toxic effects of AOX range from carcinogenic and mutagenic effects to acute and chronic toxicity and can inhibit microorganism growth (Müller, 2003; Savant et al., 2006; Deshmukh et al., 2009; Osman et al., 2013).

A further problem with PAE strengthened products is that the

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https://doi.org/10.1016/j.jclepro.2019.01.017
0959-6526/

majority of papers strengthened with epoxy thermosets cannot be recycled due to their irreversible 3-D crosslinked networks (Min et al., 2015). The used epoxy thermosets can only be incinerated or landfilled, or degraded to small molecules directly using strong acidic or basic agents (Min et al., 2015).

An ideal solution from a broad cleaner production perspective would be one that retains the required product performance while:

1. Replacing the epichlorohydrin resins, with their toxic by-products in manufacture and use, with a non-hazardous alternative.
2. Facilitating recycling.
3. Using a locally sourced bio-derived alternative.

During the last two decades, cellulose nanofibres (CNF) have emerged as a promising alternative that can meet all these requirements. Cellulose is the most abundant polymer on Earth; it is renewable, biodegradable, non-toxic and chemically versatile (Dufresne, 2013). Cellulose nanofibres are formed by breaking down cellulose fibres into their nano-scale cellulose microfibril building blocks (Lavoine et al., 2012). CNF is thus fully chemically compatible with cellulose and can be recycled with the cellulose fibres. Almost any source of cellulose can be used, including agricultural and food processing residues (Varanasi et al., 2018) as well as the fibre degradation products from paper-mill processing (Gunawardhana et al., 2017), thus utilising the principles of industrial ecology.

Furthermore CNF (Cellulose Nanofibre) is an outstanding sustainable reinforcement material (Boufi et al., 2016; Sanjay et al., 2018). Use of CNF has emerged as wet-end additive with potential applications as both a wet and dry strength agent as well as a coating to improve the barrier properties (Lavoine et al., 2012). The contribution of CNF to strength can be achieved by two mechanisms (Boufi et al., 2016). Firstly, CNF promotes the bridging between fibres which increases the fibre to fibre contact, and in turn increases the bonded area. Secondly, web-like entangled structure of CNF brings larger fibre together creating a network, which boosts the load-bearing capacity of the paper. As a result, the final paper strength is achieved by both fibre to fibre contact and the CNF network.

While CNF is a promising alternative to resin-bonded products, it must still be demonstrated that the product performance has been maintained. In this study, we investigated the development of cleaner food grade filters with the least amount of wet strength resin while delivering the same wet strength and adsorption properties as the original filters with high PAE dosage. To achieve this, a particular fibre fraction was substituted with CNF without changing the total fibre composition. Filters were prepared with a variety of PAE dosages. Performance of CNF was examined as a wet strength agent, where effect of different variables such as curing conditions and addition order of raw materials were investigated on wet strength development. Adsorption performance was examined using two different oppositely charged dye molecules. Positively charged polyelectrolytes were used to maintain the overall positive charge and adsorption characteristics of the filters.

2. Material and methods

2.1. Material

The cellulose fibres used for the composites were:

- Unrefined northern softwood bleached kraft pulp (NIST Reference Material 8495) from Procter and Gamble
- Bleached radiata pine softwood Kraft pulp refined to 400 Canadian standard freeness (CSF) in a disk refiner
- CNF prepared by processing a 0.3 wt % suspension of bleached radiata pine softwood Kraft pulp, that had been refined to 400 Canadian standard freeness, in a GEA Niro Soavi homogeniser at 800 bar pressure for 3 passes.

The fibre diameter distributions of homogenised and refined pulp were reported in our previous study (Onur et al., 2018) and are approximately 50 and 25 nm for the nanofibre fraction of the refined and homogenised pulp, respectively. Expanded perlite supplied by Dicalite Minerals Corp was used as the inorganic absorbent. Commercial PAE provided from Nopcobond Paper Technology Pty Ltd was used as wet strength resin.

Negatively charged metanil yellow (MY) and positively charged methylene blue (MB) dyes supplied from Sigma Aldrich was used to investigate adsorption characteristics. The cationic polyacrylamide (CPAM) polymer was supplied by AQUA + TECH. The F1 grade with 13 MDa molecular weight and 40% charge density was used. Polyethylenimine (PEI), branched with 10,000 Da molecular weight was purchased from Sigma Aldrich.

2.2. Methods

2.2.1. Preparation of filters

Filters were prepared by using a standard British hand sheet maker. A detailed step by step explanation of the sheet making procedure can be found in our previously published study (Onur et al., 2018). Filter suspensions were prepared by mixing pulp suspension with perlite and PAE (0.22 w/v %) or PAE & CPAM (0.1 wt %) or PAE & PEI (0.1 wt %) combinations. Filters prepared with different compositions and curing conditions are listed below.

1) 30% Fibre (1/3 refined pulp + 2/3 unrefined pulp) + 70% Perlite + 120 mg PAE-half an hour at 105 °C *(Reference sample)*
2) 30% Fibre (1/3 CNF + 2/3 unrefined pulp) + 70% Perlite + (0-5-10-30-60-90-120 mg PAE) —half an hour at 105 °C
3) 30% Fibre (1/3 CNF + 2/3 unrefined pulp) + 70% Perlite + 5 mg PAE-1 h at 105 °C
4) 30% Fibre (1/3 CNF + 2/3 unrefined pulp) + 70% Perlite + 5 mg PAE- 5 min at 150 °C *(tried by two different order of addition methods)*
5) 30% Fibre (1/3 CNF + 2/3 unrefined pulp) + 70% Perlite + 5 mg PAE & 25 mg CPAM-5min at 150 °C
6) 30% Fibre (1/3 CNF + 2/3 unrefined pulp) + 70% Perlite + 5 mg PAE & 25 mg PEI-5min at 150 °C

Suspensions were mixed using the order of addition shown in Fig. 1a, except where the effect of mixing order on wet strength development was investigated, which also used the scheme in Fig. 1b. Only the sample with 5 mg PAE sample cured at 150 °C for 5 min was tested for the effect of mixing order.

In Fig. 1a, perlite is added to CNF suspension first and then the perlite and CNF mixture is added to unrefined cellulose pulp suspension. PAE or any other positively charged polymers used with PAE for charge modification were added at the end on top of the entire suspension. Combination of PAE with other positively charged polymers was used to investigate the charge modification of the composites along with adsorption and wet strength properties.

In Fig. 1b, PAE is added to CNF mixture first and then this mixture is added to unrefined pulp suspension. Perlite suspension was then added last.

After initial air-drying, all the filters were then heated in an oven and then stored in a humidity controlled room (23 °C and 50%

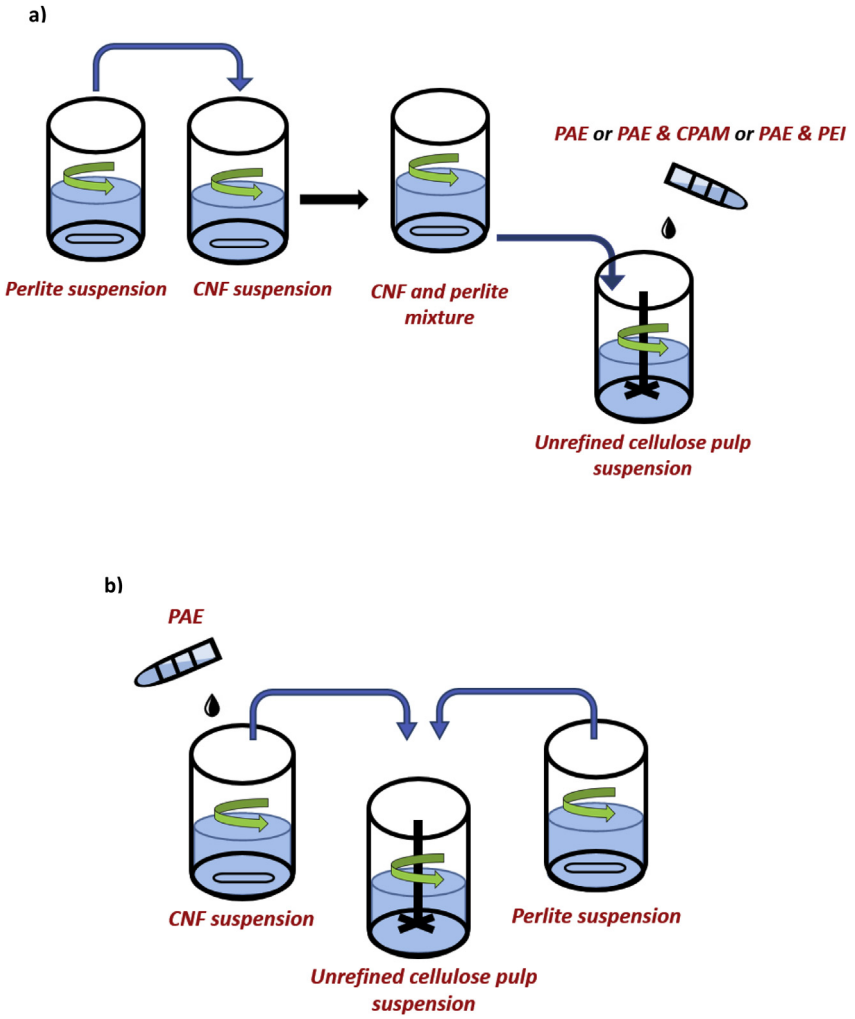

Fig. 1. Graphical representation of mixture preparation procedures.

relative humidity) for at least 24 h prior to any further use. The standard oven treatment was half an hour at 105 °C, except where otherwise noted.

2.2.2. Wet tensile strength measurements

Thickness of the composites was measured using L&W thickness tester after conditioning. An average of ten points was used to calculate the thickness of each sample. Following thickness measurements, each sample was cut into 15 mm wide strips using a sheet cutting machine. Wet strength measurements were conducted on an Instron model 5566 with a constant strain rate of 10 mm/min. At least 14 strips in total from two separately formed sheets for each sample was measured with a 100 mm test span. Sample strips were individually soaked in water for 1 min before measurement. Tensile index for each sample was calculated as tensile load per unit width divided by grammage.

2.2.3. Zeta potential

A Nanobrook Omni from Brookhaven Instruments was used to measure the zeta potential of each suspension used for making samples. A small quantity of each suspension was analysed in a cuvette cell at 25 °C before papermaking. The electrophoretic mobility was determined using laser Doppler velocimetry and zeta potential was calculated based on the Smoluchowski equation.

2.2.4. Adsorption properties

A dead-end stirred cell working with constant pressure was used to carry out adsorption experiments with 400 mL of either MY or MB dye solutions. The experimental procedure was explained comprehensively in our previous study (Onur et al., 2018). Effect of different compositions of charged polymers and CNF on charged dye molecule adsorption was investigated.

3. Results

In this section, wet tensile index of the composites was measured as a function of critical variables: PAE dosage, curing conditions and PAE adsorption methodology. Charge characteristics of the composites were investigated by zeta potential measurements. Contaminant adsorption properties were tested using an anionic (MY) and a cationic (MB) dye. Cationic polyelectrolyte addition was also examined in terms of charge modification, adsorption characteristics as well as wet strength improvement.

3.1. Wet tensile index

3.1.1. Effect of PAE dosage

Wet tensile strength properties of composites with varying PAE dosages and pulp content are presented in Fig. 2. The reference composite with refined pulp and 120 mg PAE has a wet tensile

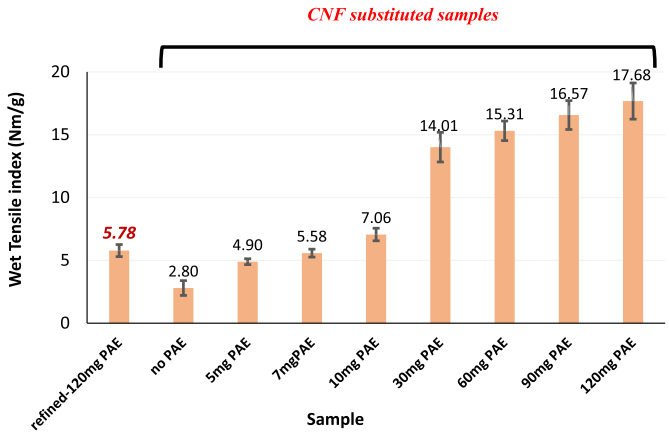

Fig. 2. Effect of PAE dosage on wet tensile index of the composites. (Curing at 105 °C for 30 min)

index of 5.78 Nm/g. Simultaneously substituting CNF for the refined pulp and eliminating PAE (Mixing procedure **(a)** in section 2.2.1 was used for sample preparation) reduced the wet tensile index to 2.80 Nm/g, showing that it is not possible to fully replace the PAE. The optimum PAE dosage with the CNF, which provides the reference wet strength, ranges between 5 mg and 7 mg under the usual curing condition and preparation technique.

3.1.2. Effect of curing conditions

The effect of curing condition was investigated with an addition level of 5 mg PAE. Samples were prepared with the procedure (Fig. 1a) given in section 2.2.1. The composites were treated at 105 °C for 1 h and 150 °C for 5 min, differing from the normal curing conditions (Fig. 3). One hour treatment at 105 °C and 5 min curing at 150 °C increased wet tensile index to 5.64 and 5.83 Nm/g, respectively. However, these differences are not statistically different from the reference material with 5.78 Nm/g. (T-test is given in supplementary material). It shows that either 1 h treatment at 105 °C or 5 min treatment at 150 °C can be selected with 5 mg PAE addition to achieve the same wet strength as the reference filter.

3.1.3. Effect of order of PAE addition

Here, PAE was added first to CNF and the mixture was then mixed with the unrefined fibres (see section 2.2.1, Fig. 1b); this differs from the previous method. Perlite suspension was added at the end and the resulting suspension was gently mixed before papermaking. Curing was set at 5 min, 150 °C. The results showed that the order of addition of resin to the fibres has no statistically significant effect (Fig. 4; with T-test analysis in supplementary material).

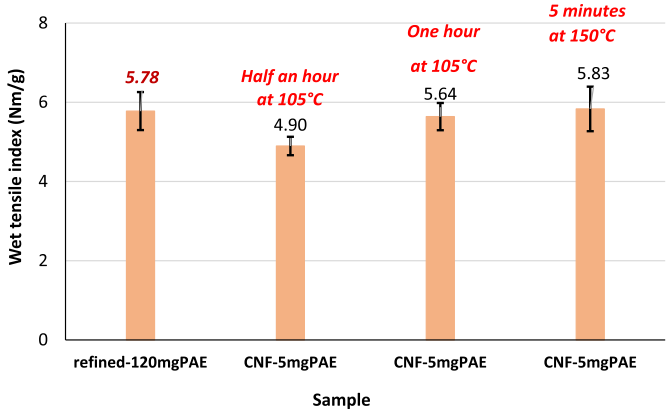

Fig. 3. Effect of curing on wet tensile index of the composites (with 5 mg PAE dosage).

3.2. Cationic polyelectrolyte addition: zeta potential and wet strength improvement

As the reduction in PAE amount also changes the charge of filter medium, charge modification was investigated with two cationic polyelectrolytes, CPAM and PEI, differing in morphology, to maintain the electrostatic adsorption properties. Sheets with CPAM and PEI were prepared by the method given in 2.1.1, Fig. 1a. Zeta potential measurements of the critical composites are given in Fig. 5. Results show that eliminating PAE dropped the electrical double layer net charge of the suspended particles from +30 mV to −37 mV, while charge is controlled from −27 mV to −15 mV by adding PAE up to 10 mg.

Substitution of 1/3 of the fibres by CNF decreased the zeta potential slightly, indicating an increase in negative charges on the surface of fibres upon homogenisation. Adding 25 mg of either CPAM or PEI with 5 mg PAE produced suspensions of zeta potential of +38 mV and +22 mV, respectively, which are similar to the reference sample.

Addition of PEI or CPAM in combination with PAE was tested to investigate the contribution of polyelectrolytes on wet strength development (Fig. 6). Adding 25 mg PEI with 5 mg PAE doubles composite wet strength. However, CPAM did not improve the wet strength significantly.

3.3. Adsorption properties

The effect of adsorbing 400 mL of anionic metanil yellow (MY) or cationic methylene blue (MB) solution was quantified on different composites. Fig. 7a and b shows the metanil yellow and methylene blue adsorption breakthrough curves, respectively. The refined-120 mg PAE composite (reference sample) has a higher adsorption capacity for MY molecules than the composite with CNF, 5 mg PAE & 25 mg CPAM (Fig. 7a). However, when CPAM is substituted for 25 mg PEI, the filter has a remarkable capacity for MY adsorption, which is even higher than that of the reference sample. Even though the zeta potential with combination of CPAM and PAE is similar to that of the reference sample, CPAM cannot

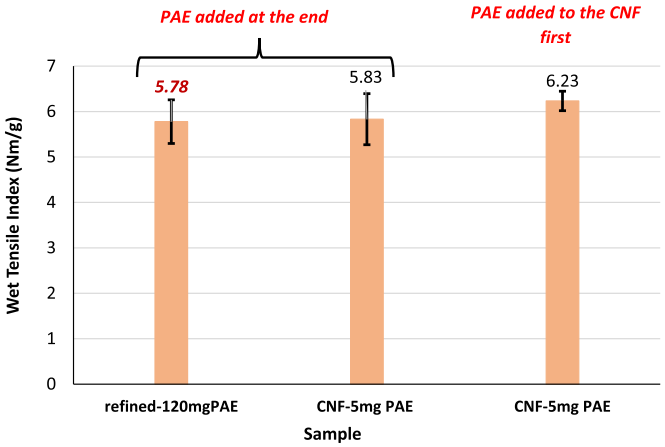

Fig. 4. Effect of order of PAE addition on wet tensile index of the composites (Curing at 5 min, 150 °C with 5 mg PAE).

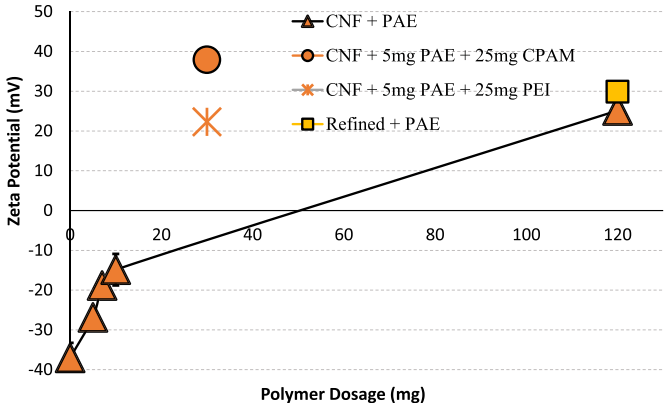

Fig. 5. Zeta potential values of composites.

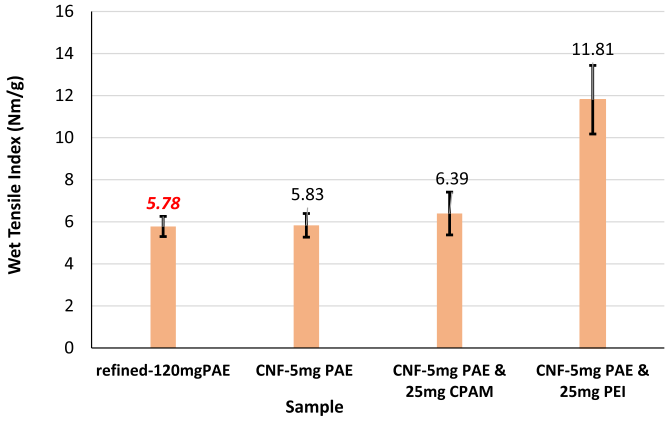

Fig. 6. Wet tensile strength properties of composites with PAE & PEI and PAE & CPAM polymer mixtures.

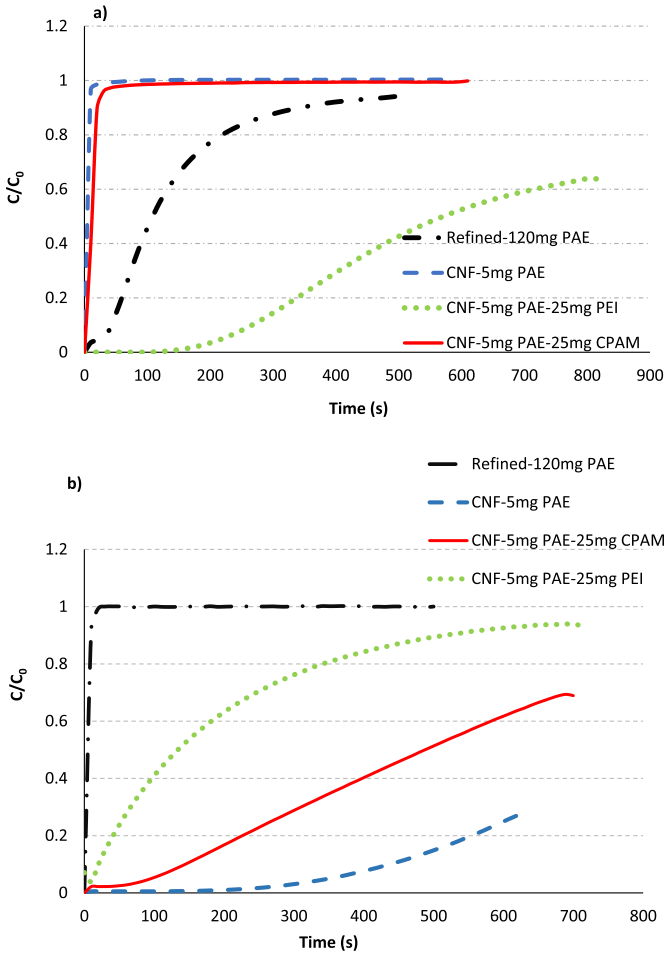

Fig. 7. Adsorption breakthrough curves of composites with (**a**) metanil yellow (**b**) methylene blue. (For interpretation of the references to colour in this figure legend, the reader is referred to the Web version of this article.)

provide the same adsorption characteristics with anionic MY. PEI addition (25 mg or less) combined with 5 mg PAE generates numerous positive charges, improving anionic contaminant adsorption onto the composites.

No additional polymer is needed for adsorbing the cationic methylene blue (Fig. 7b). The sample with 5 mg PAE and CNF provides an improved adsorption capacity shown by the breakthrough curve. Composites combining 5 mg PAE with CNF provide a remarkable separation of any cationic contaminant while retaining their original wet strength.

4. Discussion

Curing time and temperature are important variables affecting cellulosic composite wet strength at a given resin dosage. Optimising temperature and curing time can maximize the curing and crosslinking for the same amount of wet strength resin. The crosslinking of all PAE molecules on cellulose also minimizes their possible diffusion into the filtrate, an important requirement of filters in food or pharmaceutical applications. By efficient curing, either 5 min at 150 °C or 1 h at 105 °C, and substituting the refined fibre for CNF, the desired wet strength level was maintained while reducing the PAE usage by 95% (Figs. 2 and 3). This is a substantial contribution from a broad cleaner production perspective. As discussed in the introduction, CNF can be made from any cellulosic material including agricultural waste and degradation products from paper-mill processing. The straight-forward mechanical treatment we used here to produce CNF, with mechanical refining in a disk mill followed by homogenisation, does not require large factory floor area to implement and can be readily designed to any required capacity, thus allowing for on-site manufacture and use.

Adsorption in our filters is driven by electrostatic attraction between negatively and positively charged molecules. The original anionic charge of both cellulose and perlite favours the adsorption of cationic contaminants onto filters. On the other hand, cationic polymers can be adsorbed onto the fibres and perlite particles to promote anionic molecule adsorption onto filters. Charge modification is one of the reasons that PAE is used in filters in addition to the wet strength development. CPAM and PEI are the cationic polymers used in our study after PAE reduction to compensate for the loss of the positively charged PAE. Both polymers successfully restored the overall positive charge. However, there is a significant difference between the adsorption behaviour of negatively charged molecules onto samples prepared with either CPAM or PEI. CPAM is a linear and high molecular weight polymer forming flexible random coil configurations, whereas PEI is a highly branched polymer with a high charge density and much lower molecular weight. These two polymers have been previously studied and the effect of polyelectrolyte morphology on the adsorption and flocculation of microfibrillated cellulose (MFC) was analysed (Raj et al., 2016). Polyelectrolytes adsorb onto surfaces of opposite charges by electrostatic attraction. Equilibrium conformation of an adsorbed linear polymer chain is as a tails and loops configuration (Gregory and Barany, 2011). The size of the loops can be reduced if the

strength of the electrostatic interaction with the surface increases. CPAM bridges with a flattened conformation onto cellulose resulting in a smaller accessible area for negatively charged molecule adsorption. However, a branched polymer, such as PEI, adsorbs onto fibres as large rigid bridges by maintaining its shape without reconformation and flattening (Blanco et al., 2009) and leaving both the polymer and the surface more accessible to interact with the surrounding molecules. Anionic MY adsorbs onto PEI treated composites more efficiently than on CPAM treated composites; this is because most of the charges remain accessible in contrast to CPAM. PEI is a promising dendrimer to be used in conjunction with 5 mg PAE to retain adsorption characteristics. PEI also contributes to wet strength significantly (Fig. 6); a minute amount of PEI alone should be enough for both negatively charged molecule adsorption as well as wet strength development. While the contribution of cationic polymers to paper strength was already reported (Rahmaninia et al., 2018; Rice et al., 2018), the significant effect of PEI on wet strength was unexpected.

In the work here, we have shown that we can almost entirely replace PAE wet strength resin with CNF. That is, we are substituting a product with a long supply chain, a short shelf life and toxic by-products in manufacture and use with CNF, a biodegradable, renewable material which can be produced on-site from waste, thus promoting both industrial ecology and the circular economy.

This innovation also addresses the aspect of design for recycling. CNF, being composed only of cellulose is thus fully chemically compatible with micron-diameter cellulose fibres that are used to make paper. In fact, CNF has been widely investigated as a reinforcing fibre to improve the strength of conventional paper sheets (Taipale et al., 2010; Su et al., 2013), due to its outstanding web-like entangled structure, high aspect ratio and high strength (Li et al., 2016). The achievement of the required wet-strength, largely using only cellulose fibre-CNF hydrogen bonding and minimising cross-linking will allow re-dispersion in water. Thus filters wet-strengthened with CNF should be recyclable within a conventional paper recycling process, possibly with enhanced initial dispersion, because of the high wet strength.

5. Conclusions

This study investigated reducing the quantity of wet strength resin, PAE, used in cellulosic filter products by substituting a renewable, biodegradable material, cellulose nanofibres (CNF). Substituting CNF for refined fibre allowed us to reduce PAE content by 95% while retaining full wet strength. In addition, the use of PEI to substitute for the cationic charge lost upon removal of the PAE was found to allow for the adsorption of both negatively and positively charged contaminants, in comparison to filters prepared with PAE, which only adsorbed negatively charged contaminants. This substantial reduction in PAE usage is particularly interesting for cellulosic filters used in beverage filtration applications. PAE is used as an indirect food additive in these type of filter products and the allowed concentration is very limited. Therefore, CNF substitution is a promising strategy for high performance cellulose fibre based filters while reducing PAE usage by 95%, drastically reducing AOX generation in PAE production and application. CNF's biodegradable and renewable nature provides cleaner products by significantly reducing the chemical impact on the environment and should be further investigated for any paper product currently using PAE to develop the required wet-strength.

Acknowledgements

This work was supported by the Chemicals and Plastics Manufacturing Innovation Network and Training Program (C&P GRIP), Victorian Government, Monash University and 3M Australia.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2019.01.017.

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