EFFECT OF CALCIUM HYDROXIDE FILLER LOADING ON THE PROPERTIES OF BANANA STEM HANDSHEETS

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Calcium hydroxide filler dispersions, of various particle sizes, were prepared by mixing sodium hydroxide with calcium chloride, in various concentrations, at room temperature. The resulting filler dispersions were added, in various amounts, to the banana stem mechanical pulp, which was then converted to handsheets. Increasing the filler loading increased the tensile index but reduced the tear index and water absorption of the handsheets. The SEM micrographs of the handsheets surfaces and the tensile fractured surfaces of the handsheets tensile test specimens showed that increasing the filler loading resulted in the formation of more and bigger filler aggregates in the spaces between the fibre. The filler particle size did not have any significant effect on the handsheets properties.

Keywords: Paper making; Banana stem; Filler; Calcium hydroxide; in-situ reaction; Water absorption; Tensile strength

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INTRODUCTION

The utilization of natural fibre in many industries has produced many exciting possibilities. Natural fibre is normally obtained from wood, but due to the awareness of environmental problems associated with deforestation, people are looking for alternative sources of raw materials to obtain natural fibres. Advances in research and technology have shown that favorable alternative sources of the raw material for natural fibre are annual crops or agricultural wastes (Cordeiro *et al.* 2004). The outcome of this is increased utilization of agricultural wastes such as rice straw, bagasse, wheat straw, and banana to improve the structure performance of the products (Oliveira *et al.* 2008; Thomas and Pothan 2008).

Banana plant is widely planted in many tropical countries, and India is the largest producer of banana in the world, with 16.8 million tons per year, followed by Ecuador and Brazil with 5.4 million and 5.3 million tons per year, respectively (Ajayan *et al.* 2003). At present, the banana fibre is considered a waste product of banana cultivation and is not properly utilized. The banana trunk or banana stem is normally left in the plantation to be converted via vermicomposting to vermicast, which is used as organic fertilizer. Banana stem contains a high amount of cellulose, which is about 67.34% of its total weight (Cordeiro *et al.* 2004). Basically, banana fibres are preferred in pulp and paper industries because they possess good physical and mechanical strength and are low cost and low weight. It has been shown that greaseproof paper made from hydrated banana pulp can have a tensile index of 51.20 N mg^{-1}, burst index of 6.21 kg pam^2g^{-1}, tear index of 7.00 mNm^2g^{-1}, and a good blister and oil resistance (Goswami *et al.* 2008).

Normally in the packaging industries, most of the commercially available paper and paperboard are produced according to the market demands that are based on the choice of fibre (either bleached or unbleached, chemically or mechanically separated, virgin or recovered fibre), the treatment, and additives used at the stock preparation (Kirman 2005). Recently, due to increased environmental awareness, researchers have started to turn a new page for paper production by substituting toxic and non-ecofriendly chemicals to produce green paper production. In this study, mechanical pulping is mostly preferred, since it does not consume any chemicals that may have serious adverse effects on the environment. Furthermore, mechanical pulping produces higher yields compared to other methods of pulping. Unfortunately there are some drawbacks to using mechanical pulp. In paper making industries, the addition of filler into the paper stock has been practiced since the ancient days to improve the opacity, brightness, and printability of the paper product. In some instances, filler is added to lower the production cost of paper, because the cost of filler is often less than the cost of the fiber. The most widely used fillers are finely divided white minerals such as calcium carbonate, kaolin clay, titanium dioxide, and magnesium silicate that serve to fill the voids that exist between the fibre (Smook 1992). The use of these mineral fillers always results in a denser, more opaque but weaker paper because the strength always is reduced at higher filler loading (Hu *et al.* 2009). Advancement in paper technology has resulted in the utilization of specialty fillers for the production of papers with special properties such as antimicrobial paper (Kim *et al.* 2005), deodorant paper (Shen *et al.* 2010), flame retardant paper (Shen *et al.* 2009), and magnetic paper (Zakaria *et al.* 2004a,b).

Lately, due to increased emphasis on green and environmentally friendly paper-based products, researchers are focusing on finding methods to produce water repellent paper without coating. Many researchers have proved that internal sizing may produce paper that is able to resist water or other fluids (Hubbe 2006). The action of rosin-alum sizing systems has served the paper industry's needs for making effective water repellent papers (Herbert 1990). The hydrophobic group in alkylketene dimer (AKD) and alkenylsuccinic anhydride (ASA) helps to explain the paper's improved resistance to wetting (Hubbe 2006). Some researchers also have reported that incorporation of filler such as calcium hydroxide can give the water repellency effect. Calcium hydroxide is used in water repellence applications such as in concrete and in other areas. In concrete industries, calcium hydroxide also acts as water-repellent material by lining the pores of the concrete (Egnilton 1987). In the UK, calcium hydroxide is recognized as a cavity-based material for composite resin restoration (Papadakou *et al.* 1990). Furthermore, in dentistry, calcium hydroxide is also being used as filler in root canals. However, even though calcium hydroxide is widely used as additive in many products, very little information has been published on the use of calcium hydroxide as filler in paper.

The aim of this research was to study the potential of calcium hydroxide filler as reinforcement for banana stem handsheets for packaging applications, especially in increasing the paper strength and reducing the water absorption of the paper. In this work, a calcium hydroxide ($Ca(OH)_2$) dispersion was prepared by mixing sodium hydroxide (NaOH) and calcium chloride ($CaCl_2$) stoichiometrically. The resulting filler, which is calcium hydroxide, was then incorporated into banana stem fibre by mixing it with pulp stock, and the mixture was converted to handsheets. The relevant handsheet properties

such as physical and mechanical properties were measured, and the effect of calcium hydroxide loading on the handsheet properties was investigated.

EXPERIMENTAL

Materials

Sodium hydroxide and calcium chloride were purchased from Sigma-Aldrich Co. Ltd., and banana trunk was taken from the garden of the School of Art of Universiti Sains Malaysia. A commercial brown paper product having a basis weight of 60 g/m^2 was used for comparison purpose in this research; it was purchased from Zarm Scientific and Supplier Company.

Methods

Preparation of calcium hydroxide ($Ca(OH)_2$) dispersion

$Ca(OH)_2$ dispersions of different concentrations were prepared by mixing appropriate amounts of NaOH and $CaCl_2$ at room temperature with different concentrations of NaOH and $CaCl_2$, as shown in Table 1. The mixing was done in a 500 mL beaker, and the resulting mixture was stirred for 30 minutes to ensure proper mixing. Then the mixture was left for a while before mixing it into the pulp stock.

Table 1. Concentrations of Chemical Used to Prepare Calcium Hydroxide Dispersions

Condition	NaOH concentration	$CaCl_2$ concentration
1	0.5M	1M
2	1M	0.5M
3	1M	1M

Measurement of filler particle size of calcium hydroxide filler

The mean diameter was used as the measure of the particle size and was determined by using the Laser Diffraction Particle Size Analyzer (Malvern Mastersizer 2000) supplied by Malvern Instrument Ltd.

Preparation of banana fiber

Banana stem was cut into small pieces and washed with tap water. Then the banana stem was refined using a Sprout Bauer Refiner with 85˚ plate gap to ensure complete separation of fiber. The resulting pulp was screened using the Sommerville Flat Screen with 0.15 mm ± 0.005 mm openings in the screen to separate the coarse and fines fibers.

Preparation of handsheet

About 24 g of oven-dried (OD) banana stem mechanical pulp was weighed into a 2500 mL plastic beaker, and about 2000 mL of distilled water was added to reach 1.2% consistency. Then $Ca(OH)_2$ dispersion was added into the pulp stock, and the amount added was varied according to 10%, 20%, 30%, 50%, and 70% based on the amount of pulp used (OD). The mixture was then mixed using a standard disintegrator at 10,000

revolutions to make them homogeneous. After that, the consistency of the stock was adjusted to 0.3% by adding the appropriate amount of distilled water. The pH of the pulp stock was measured using a pH meter. Handsheets were made using the handsheet making machine according to TAPPI method T-205sp-02, with the target basis weight of 60 g/m^2. The resulting handsheets were dried in a standard condition room at 50.0 ± 2.0% RH at 23˚ ± 1˚C as specified in TAPPI method T402om-93. After that, the handsheets were allowed to dry completely in the conditioning room for 24 hours at room temperature.

Tests

Water absorption of the handsheets was measured using the Cobb60 test. The tensile index was measured according to TAPPI 404cm-92, using the Lloyd tensile testing machine, whilst the tearing index was done according to TAPPI 414om-04 test method using the Elmendorf tearing machine.

Filler content

The filler retention was used as the measure of filler content of the handsheets. The filler retention content was determined by measuring the ash content of the hand-sheet according to TAPPI 413om-06. The sample was placed in a crucible and ignited in a muffle furnace at 525 ± 25 ˚C. When the sample was completely combusted, which was indicated by the absence of black particles, the sample was allowed to cool in a desiccator, and the filler retention was calculated according to the formula below:

$$\text{Filler retention (\%)} = \frac{x1-x2}{x3\ (1-x4)} \times 100 \quad (1)$$

where *x1* is the ash content of the handsheet, *x2* is the ash content of the blank handsheet (handsheet without filler), *x3* is the filler loading (%), and *x4* is the ignition loss of the filler (%).

Morphology

A small portion of the handsheets was cut, and the top surface was coated with a thin layer of gold. Then the sample was scanned using the LEO Supra 50 Vp Field Emission Scanning Electron Microscope (FESEM), obtained from Carl Zeiss SMT, Oberkochen, Germany.

RESULTS AND DISCUSSION

Effect of Chemical Concentrations on the Particle Size of Filler

Figure 1 shows the average particle size of the filler obtained from different concentrations of chemicals. Reacting 0.5 M of NaOH with 1 M of $CaCl_2$ produced filler particles having diameters of around 1.02 µm. Increasing the concentration of NaOH or $CaCl_2$, however, produced filler of bigger diameter, as evident from the results of reacting 1M NaOH with 0.5M $CaCl_2$ and 1M NaOH with 1M $CaCl_2$.

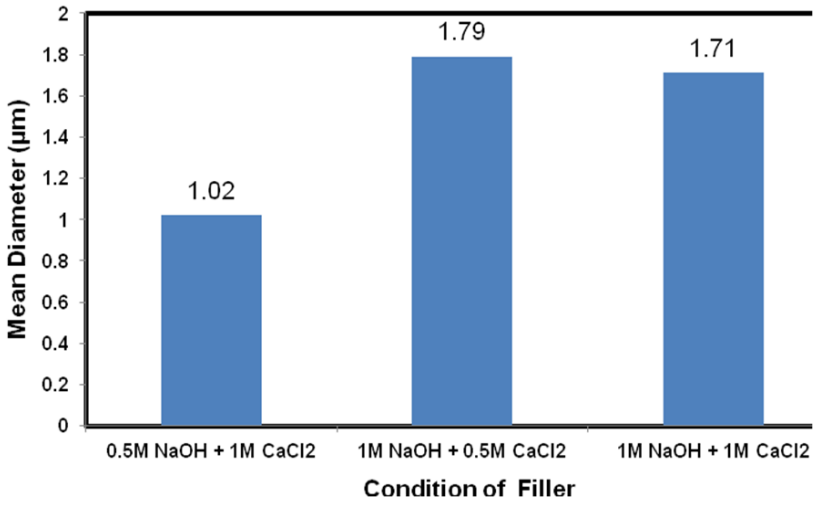


Fig. 1. Particle size of filler prepared by different concentrations: a) 0.5M NaOH + 1M $CaCl_2$, b) 1M NaOH + 1M $CaCl_2$, and c) 1M NaOH + 1M $CaCl_2$

Filler Retention

The retention of calcium hydroxide filler of different particle sizes in the hand-sheets is shown in Fig. 2. The retention efficiency of filler in paper making strongly influences the production cost, cleanliness of the paper making system, and the load of pollution at the disposal system (Vengimallla *et al.* 1999). We can see that the retention of the smaller particles was lower than the big particles, and the amount of filler retained increased with filler loading. At 70% filler loading, around 35% of the filler was retained in the handsheets. During handsheets making, some of the filler particles were lost due to the pressure applied. The filler particles, especially the smaller ones, can pass through the spaces between the fibre in the handsheets.

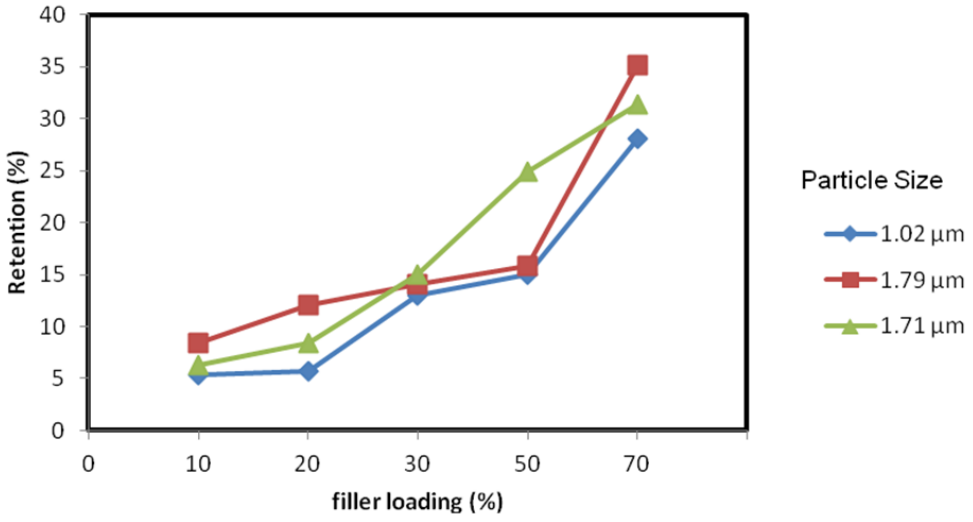


Fig. 2. Filler retention in the handsheets with different filler loading

The increase of filler retention with filler loading can be attributed to the formation of filler aggregates. Since the size of the aggregates is much bigger than the size of the individual particles, more filler aggregates will be retained in the handsheets.

It has been reported that at high pH, flocculation of fibre and filler particles will take place and this will result in high filler retention (Casey 1981; Jaycock and Pearson 1975). In this study, however, the effect of pH on the filler retention was not observed, as shown in Fig. 3. The pH of the pulp stock was around 9, but when dispersion of calcium hydroxide was added the pH increased to 13. However, when more calcium hydroxide was added to the pulp stock, the pH remained at 13 but the retention increased from around 10% to nearly 35%.

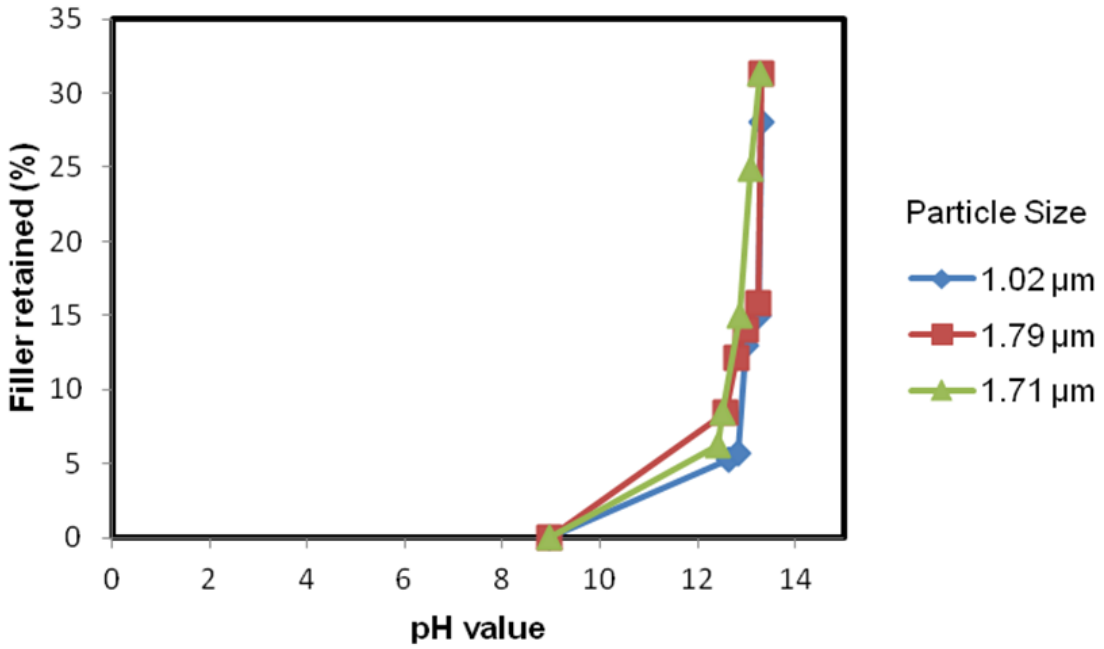


Fig. 3. Effect of pH on the filler retention

Tensile Index

In paper technology, tensile index is normally used as a parameter to describe and compare the fibre-to-fibre bonding properties in handsheet (Savcor 2007). From Fig. 4, it could be seen that tensile index increased with filler content. Filler content is used to represent the amount of filler retained in the handsheets. At higher filler content, tensile index seemed to drop a little, but the values are still higher than that of the handsheets without filler. The effect of particle size on the tensile index was variable, even though we observed some increase in the handsheet containing calcium hydroxide filler of average particle size of 1.71 µm. Also, the tensile index of the handsheets was observed to be higher than the tensile index of the commercial paper.

The results shown in Fig. 4 are in contradiction with the observations made by other researchers (Shen *et al.* 2010). Usually incorporation of inorganic filler in paper has resulted in the reduction of tensile index because of the disruption of the fiber-fiber bonding in the paper. During deformation, as in the tensile test, the load could not be

transferred effectively from fibre to fibre, and this resulted in the reduction of the tensile index of the handsheets.

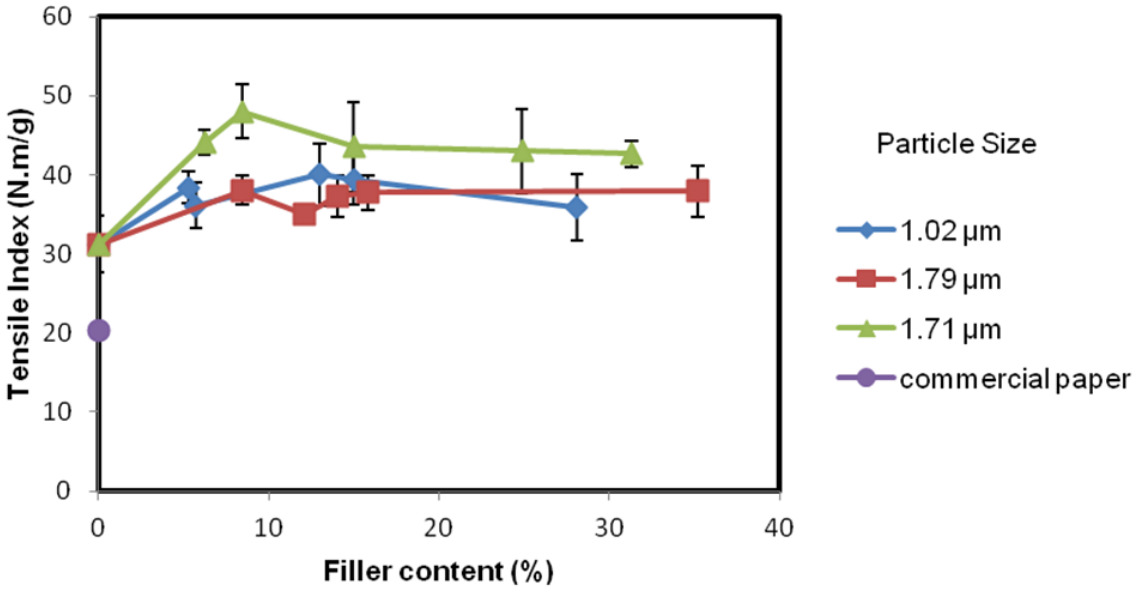
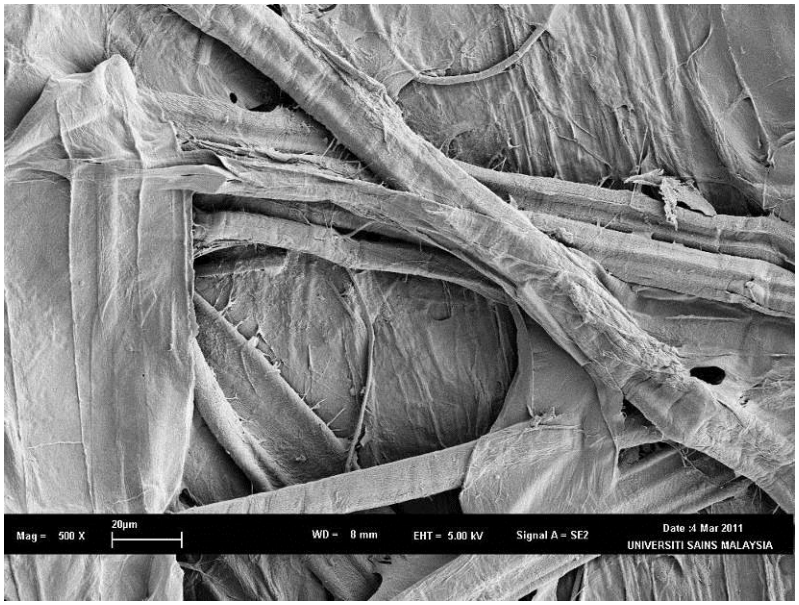


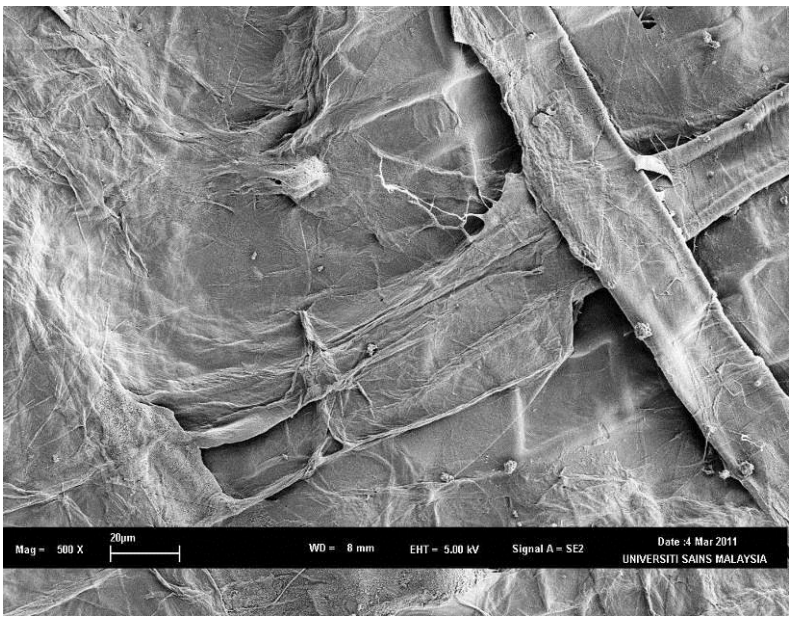
Fig. 4. Effect of percentage filler content and filler particle size on the tensile index of handsheets

A possible reason for the increase in the tensile index could be explained by analyzing the SEM of the handsheets and the tensile fracture surfaces of the tensile test specimen (Figs. 5, 6, and 7).



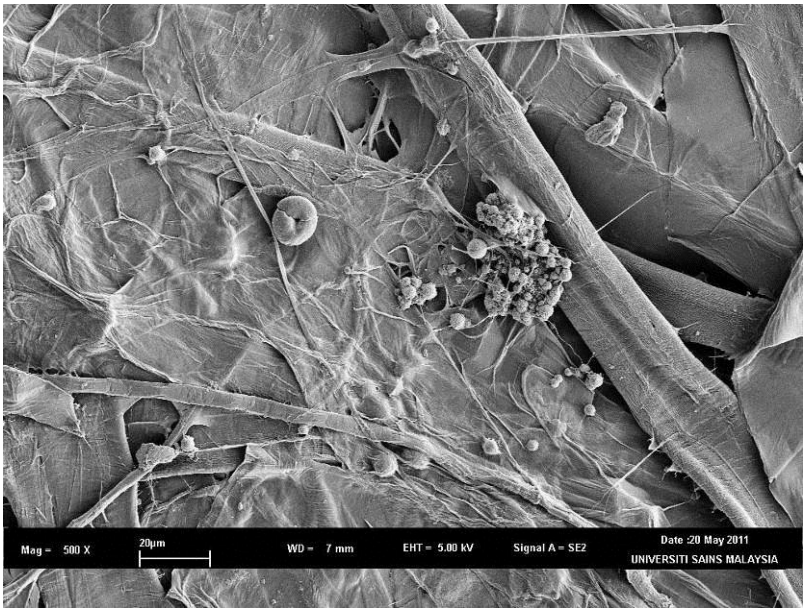
(a)

Fig. 5a. SEM image of handsheet surface with (a) 0% filler content, (b) 5% filler content, (c) 10% filler content, (d) 15% filler content, (e) 25% filler content and (f) 35% filler content, respectively



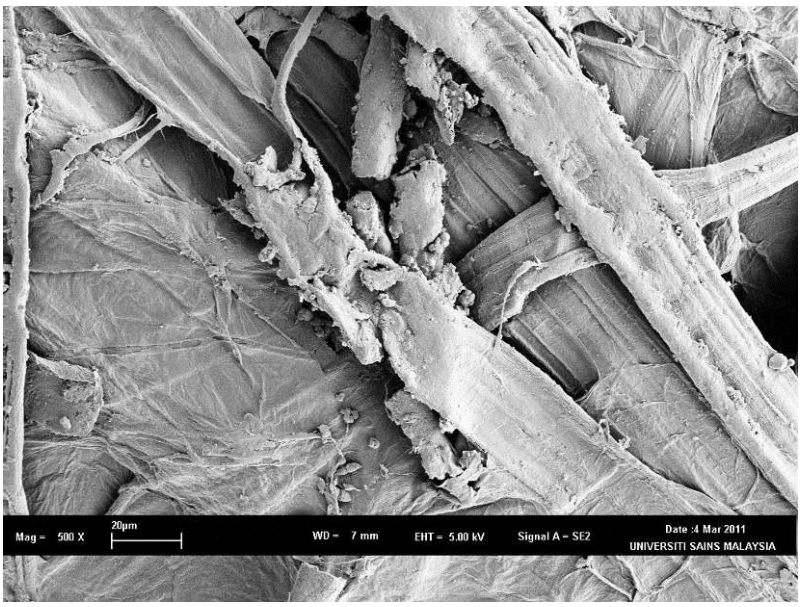
(b)

Fig. 5b. SEM image of handsheet surface with (a) 0% filler content, (b) 5% filler content, (c) 10% filler content, (d) 15% filler content, (e) 25% filler content and (f) 35% filler content, respectively



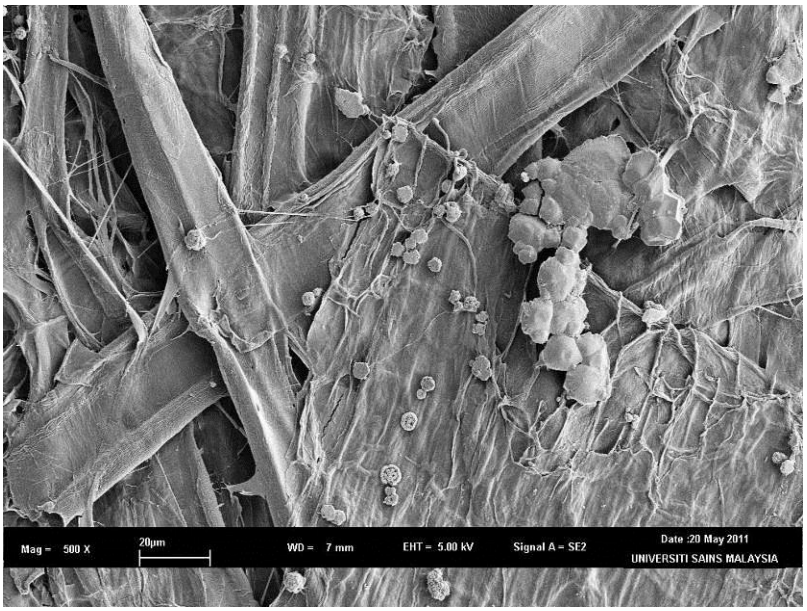
(c)

Fig. 5c. SEM image of handsheet surface with (a) 0% filler content, (b) 5% filler content, (c) 10% filler content, (d) 15% filler content, (e) 25% filler content and (f) 35% filler content, respectively



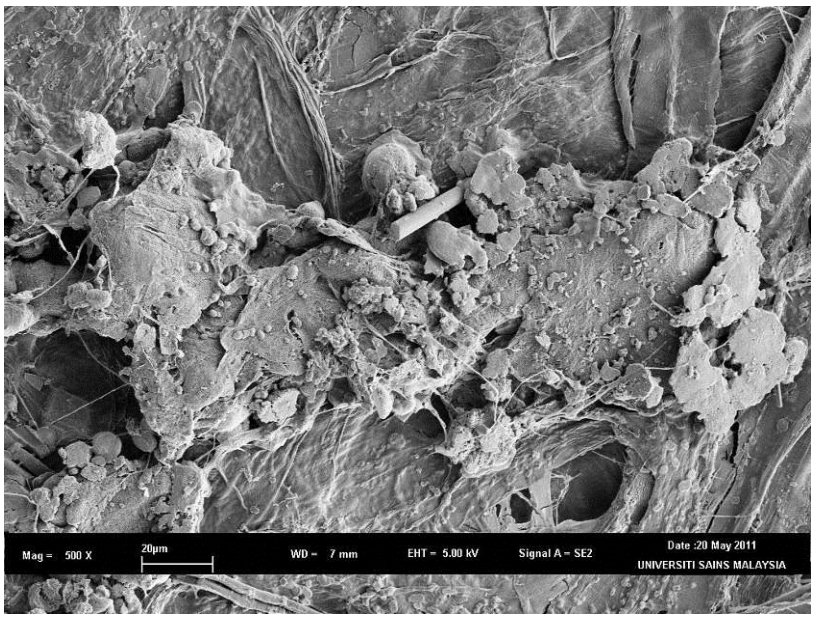
(d)

Fig. 5d. SEM image of handsheet surface with (a) 0% filler content, (b) 5% filler content, (c) 10% filler content, (d) 15% filler content, (e) 25% filler content and (f) 35% filler content, respectively



(e)

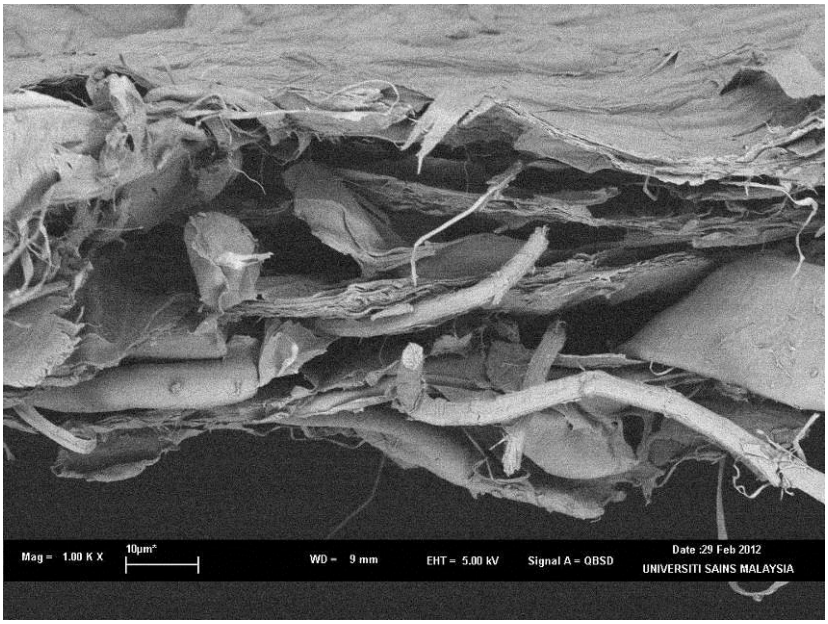
Fig. 5e. SEM image of handsheet surface with (a) 0% filler content, (b) 5% filler content, (c) 10% filler content, (d) 15% filler content, (e) 25% filler content and (f) 35% filler content, respectively



(f)

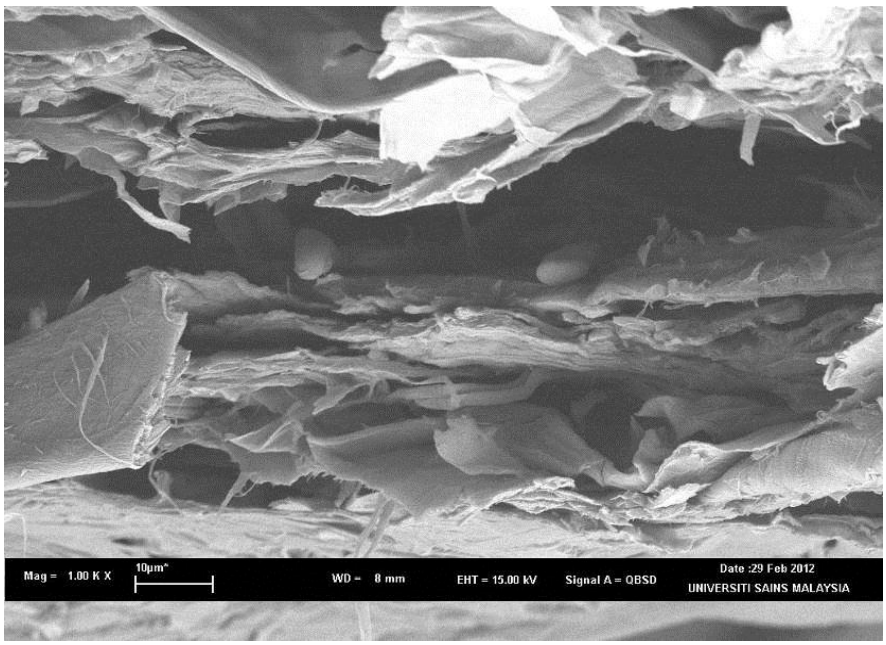
Fig. 5f. SEM image of handsheet surface with (a) 0% filler content, (b) 5% filler content, (c) 10% filler content, (d) 15% filler content, (e) 25% filler content and (f) 35% filler content, respectively

From Fig. 5, it could be seen that some filler particles attached themselves on the fibers and some formed aggregates in the areas between the fibers. The number and size of these aggregates increased with filler content and at higher filler content, these aggregates seemed to fuse together to form a continuous layer of filler.



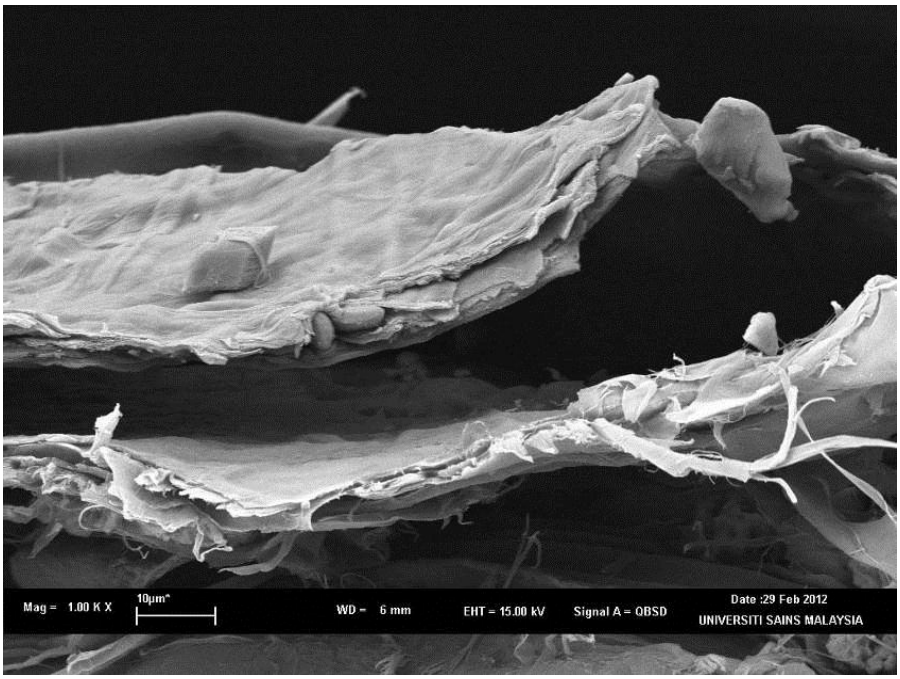
(a)

Fig. 6a. SEM images of tensile fracture surface for hansheet with a) 0%, b) 5%, c) 10%, d) 15%, e) 25%, and f) 35% filler content



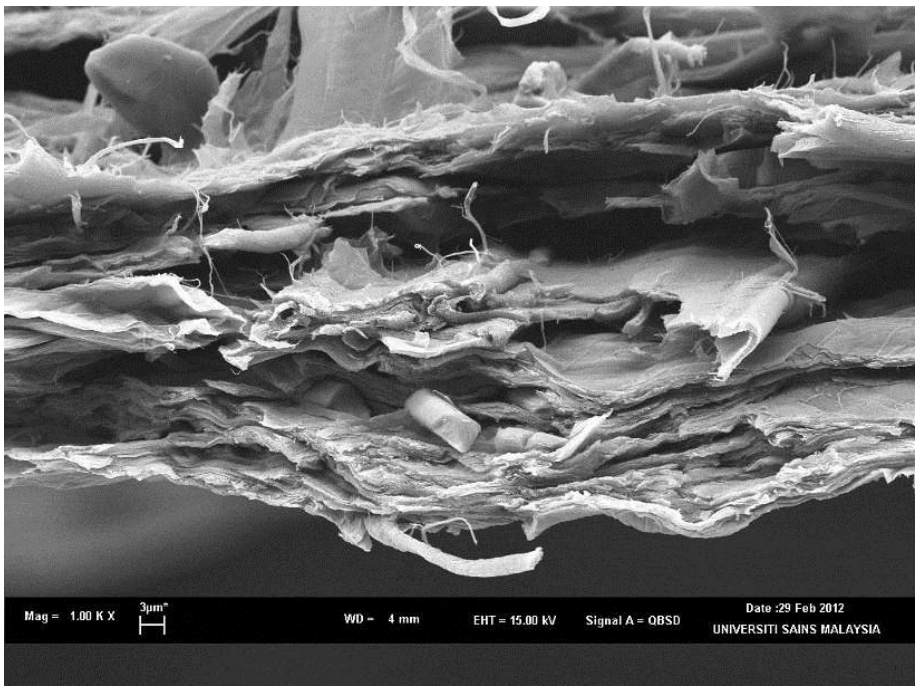
(b)

Fig. 6b. SEM images of tensile fracture surface for hansheet with a) 0%, b) 5%, c) 10%, d) 15%, e) 25%, and f) 35% filler content



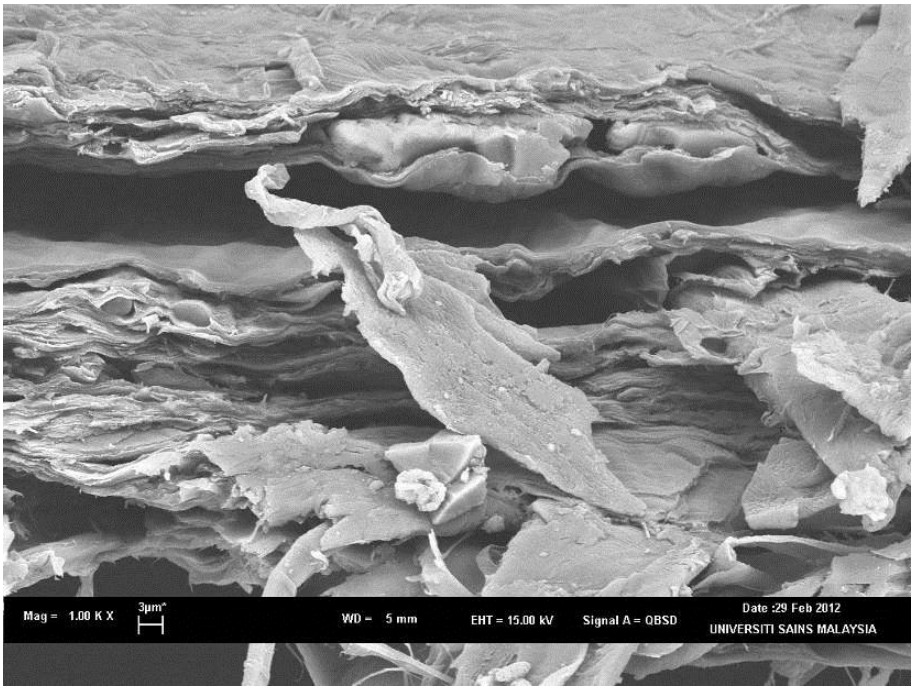
(c)

Fig. 6c. SEM images of tensile fracture surface for hansheet with a) 0%, b) 5%, c) 10%, d) 15%, e) 25%, and f) 35% filler content



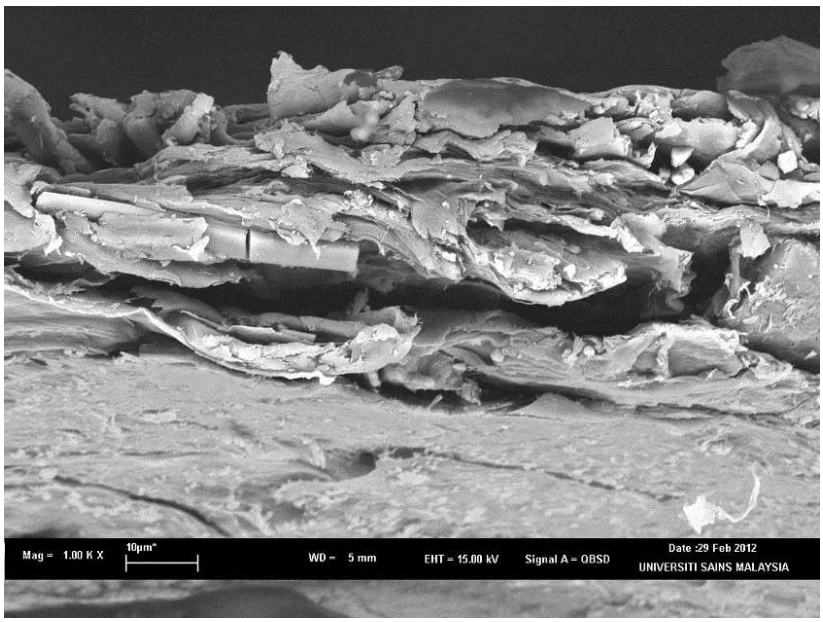
(d)

Fig. 6d. SEM images of tensile fracture surface for hansheet with a) 0%, b) 5%, c) 10%, d) 15%, e) 25%, and f) 35% filler content



(e)

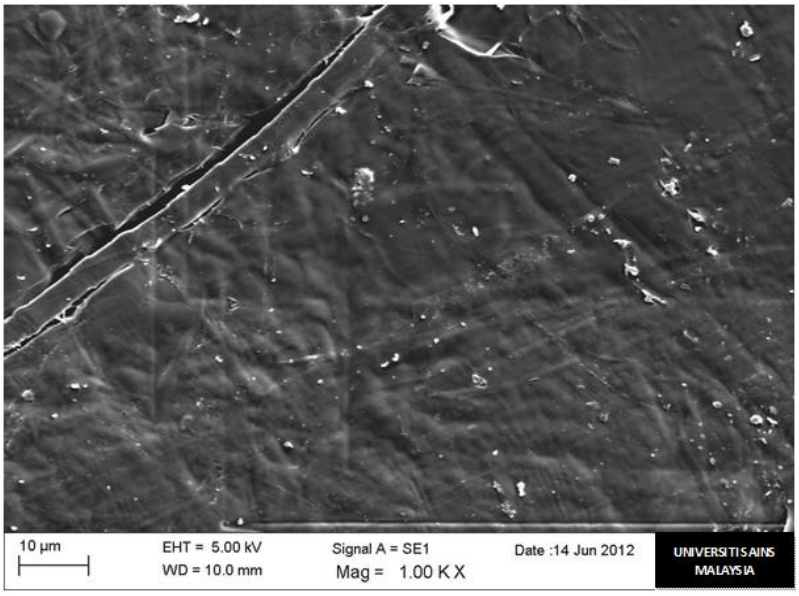
Fig. 6e. SEM images of tensile fracture surface for hansheet with a) 0%, b) 5%, c) 10%, d) 15%, e) 25%, and f) 35% filler content



(f)

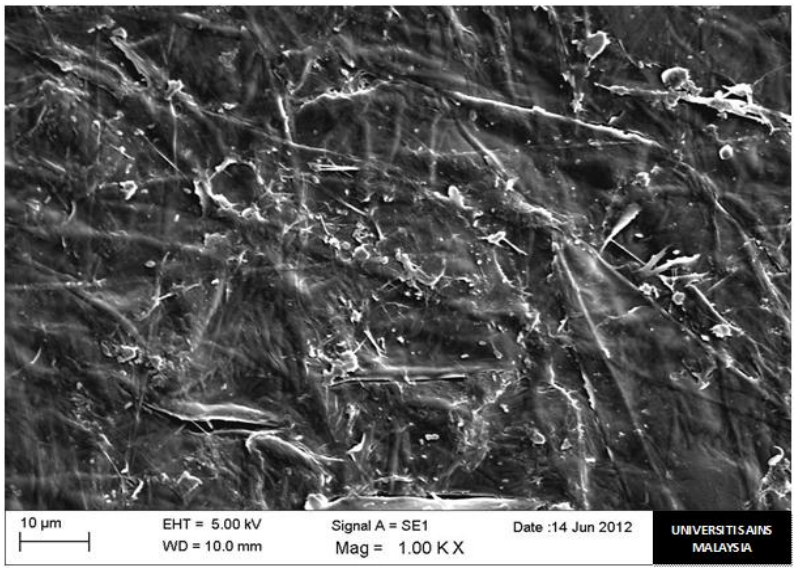
Fig. 6f. SEM images of tensile fracture surface for hansheet with a) 0%, b) 5%, c) 10%, d) 15%, e) 25%, and f) 35% filler content

Based on Fig. 6 which shows micrographs of tensile fractured surfaces of the handsheets test specimens, it can be seen that there were no filler aggregates formed inside the hand-sheets. Filler aggregates were formed on the bottom surface of the handsheets, Fig. 7.



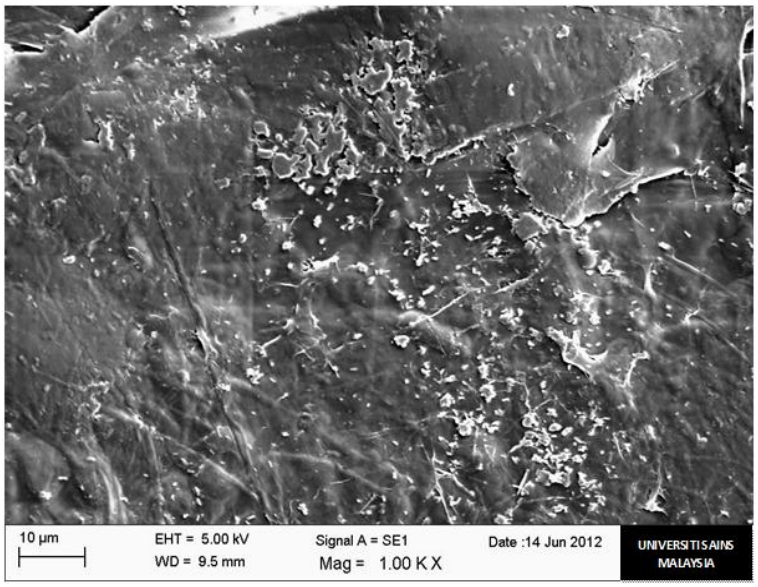
(a)

Fig. 7a. SEM images of bottom surface of the handsheet with a) 5%, b) 15% and c) 35% filler



(b)

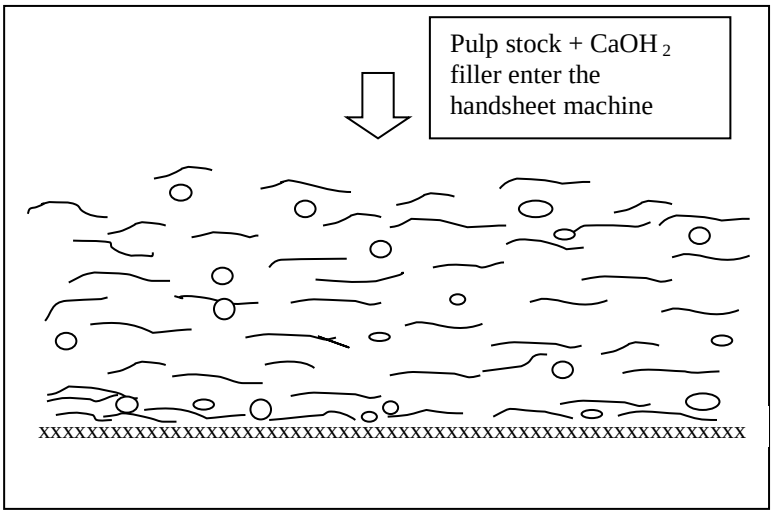
Fig. 7b. SEM images of the bottom surface of the handsheet with a) 5%, b) 15% and c) 35% filler



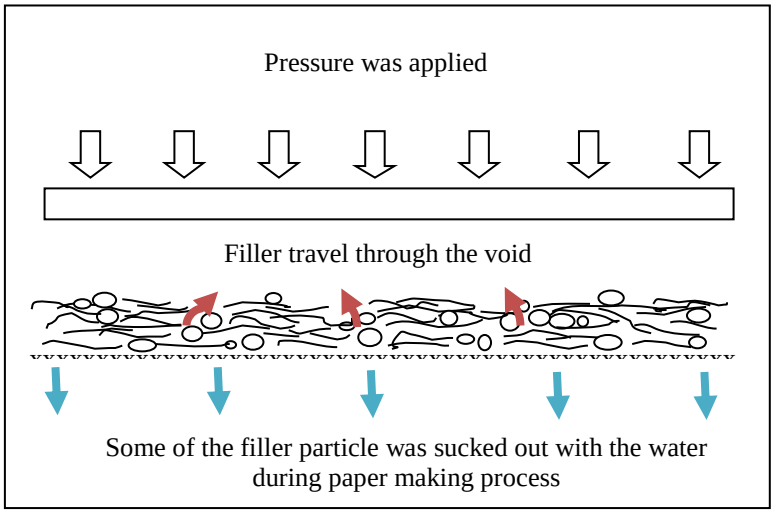
(c)

Fig. 7c. SEM images of the bottom surface of the handsheet with a) 5%, b) 15% and c) 35% filler co

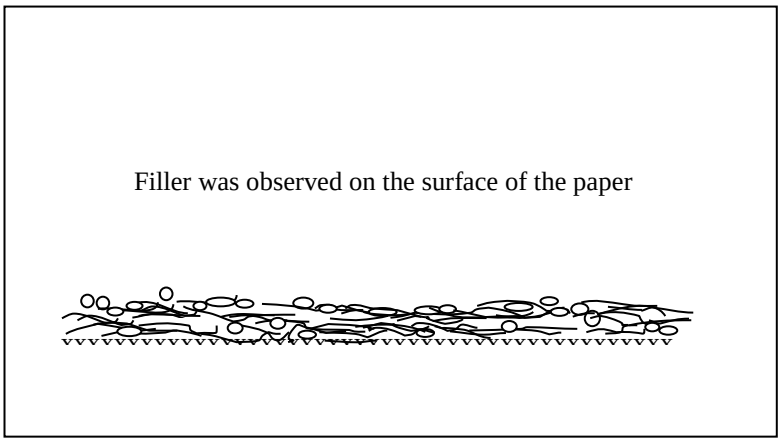
The reason why the aggregates were formed on the bottom surface and not inside the handsheets, is that the filler particles were sucked out of the handsheets when pressure was applied during the formation of the handsheets, as mentioned previously. The formation of filler aggregates on the surface of the handsheets had little effect on the fiber-fiber bonding inside the handsheets, and thus the tensile index was not affected too much with the increase in filler content. The mechanism of the formation of filler aggregates on the surface of the handsheets could be explained by Fig. 8.



Before pressing



During pressing



After pressing

Fig. 8. Illustration describing the movement of filler before, during, and after pressing process

During pressing, the filler particles were sucked out from the handsheets together with the water. Most of the particles were sucked out to the bottom surface of the handsheets, and some of the particles occupying the top layer were squeezed out to the top surface of the handsheets during pressing. During pressing, the particles traveled out of the handsheets through the spaces or voids between the fibres, and as they emerged from the handsheets, these particles settled and occupied the areas between the fibers, on the surfaces of the handsheets, in the form of aggregates. As more pressure is applied, these aggregates were squeezed further and became deformed and flattened to form a continuous layer of filler. This explains the phenomena observed in the SEMs shown in Figs. 5 and 7.

Tear Index

The tearing index of the handsheets was lower than the commercial paper, and it decreased with increasing calcium hydroxide content, as shown in Fig. 9.

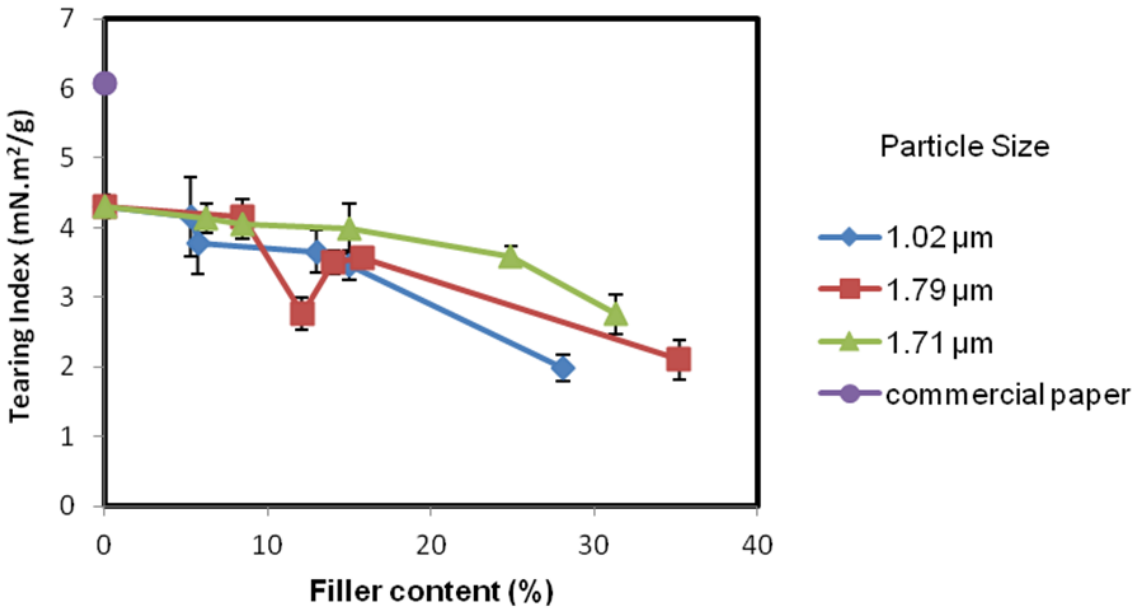


Fig. 9. The change of tearing index of handsheets with filler content and filler particle size

The tearing resistance of handsheets is normally dependent on the fibre strength, which in turn is related to the fibre length and the fibre flexibility. Those criteria are the most important factors that affect the tearing index (Casey 1980). The tearing index of the handsheets was lower than that of the commercial paper because the pulp was made using the mechanical pulping method which produced shorter fibre, as shown in Table 2.

Shorter fibre produced less flexible handsheets. As shown in Table 2, the average length of the fibre was 0.61 mm, and the percentage of the longest fibre was just 2.2%. More fibre was found in the range of medium length and short length, which was about 48.30% and 30.78%, respectively. The reduction of tearing index with filler content was due to increased handsheets stiffness, which reduced the flexibility of the handsheets

(Casey 1981). Another reason for the reduction in the tearing index was due to the reduction in the fibre to filler ratio in the handsheets, as explained by Shen *et al.* 2010.

Table 2. Characteristics of Pulp Fibre

Item	Characteristic
Average fibre length	0.61 mm
Length weighted average	0.95 mm
Average debris length	1.28 mm
% of fines	13.69%
% of short length fibre	30.78%
% of medium length fibre	48.30%
% of long length fibre	2.20%

The tearing index of the handsheets were not dependent on the size of the filler particles.

Water Absorption

Figure 10 shows the effect of filler content and filler particle size on the water absorption of the handsheets.

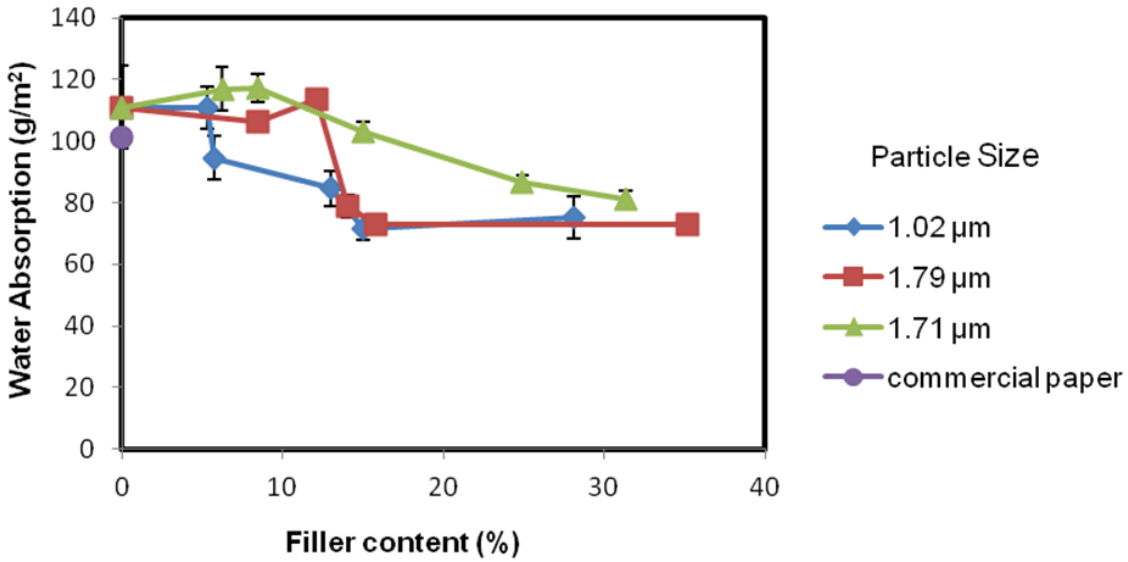


Fig. 10. Effect of filler content and filler particle size on the water absorption of the handsheets

The water absorption of the unfilled handsheets was higher than that of the commercial paper. Increasing the percentage of filler content decreased the water absorption, and handsheets containing more than 15% of filler content absorbed less

water than the commercial paper. The effect of particle size on water absorption, however, was variable. The reduction of water absorption with filler content was due to the formation of filler aggregates on the surfaces of the handsheets, as shown in Fig. 5. It seemed that as more filler was added to the pulp, more big aggregates were formed on the surface. These aggregates covered the spaces between the fibre, Fig. 5(f), and thus reduced the water absorption, since water was absorbed through these spaces. This observation was in line with the work reported by Topgaard and Söderman (2001) and Singh *et al.* (2009).

CONCLUSIONS

1. The particle size of the calcium hydroxide filler produced was dependent on the concentrations of the NaOH and CaCl used. Larger particles were produced with higher concentration of the chemicals. The addition of $Ca(OH)_2$ modified the mechanical and physical properties of the handsheets.
2. The tensile index of the handsheets increased with filler content, but the tear index and water absorption showed the opposite trend. More and bigger filler aggregates were formed as filler content was increased, and these filler aggregates covered the spaces between the fibre, which resulted in the reduction of water absorption.
3. The effect of filler particle size on the mechanical and physical properties of the handsheets was variable.

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