Effect of Surface Sizing of Black Liquor on Properties of Corrugated Medium

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Spent cooking liquor from kraft pulping, known as black liquor, was applied to linerboard (corrugated medium) by surface sizing in order to increase strength properties. The influence of alum in black liquor on linerboard properties was also investigated. The surface application of black liquor improved the dry strength of linerboard. A slight addition of alum into black liquor (2.5% on black liquor solids) significantly improved strength properties such as tensile strength, TEA, compressive strength, and bursting strength. The results implied that black liquor with alum can be used for industrial grade papers that require high strength properties. The air permeability of corrugated medium increased after 5 g/m^2 of dry pick-up. Black liquor appeared to penetrate the paper pores, and aggregates of lignin and carbohydrates in the black liquor increased bonds between fibers, which improved strength. The surface sizing of black liquor and alum addition also affected the hydrophobicity of linerboard.

Keywords: Black liquor; Surface sizing; Corrugated medium; Dry strength; Alum; Lignin

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INTRODUCTION

Corrugated board, or containerboard, is a packaging material for manufacturing corrugated boxes. In its simplest form, single-wall corrugated board consists of a fluted corrugated sheet (corrugating medium) and two linerboards (top and bottom liners). The corrugated material is sandwiched between two liners (Kiviranta 2000). In many cases, the corrugating medium and the test liner (bottom liner) have been generally produced from relatively low quality waste paper (OCC, old corrugated container) to reduce the production cost (Lee *et al.* 2000; Yoon and Sung 2015). This would also decrease strength properties of linerboard. However, the consumer's demand for higher strength linerboard has been increasing. Hence, the issue to improve the quality or strength of linerboard from the cheaper and lower quality law materials is becoming increasingly important.

One approach is to apply dry strength aids such as starches or resins by surface treatment or by wet end addition (Jeong *et al.* 2012; Lee *et al.* 2015; Jang and Park 2016; Seo *et al.* 2016). Kim *et al.* (2012) suggested impregnating linerboard with PVAm (polyvinylamine) to improve strength properties, especially wet tensile strength. The recycled fibers from cotton clothing waste could also be used with OCC fibers to improve the strength properties of linerboard, especially folding resistance (Hong *et al.* 2014). Saidan (2013) reported that heat treatment of linerboard at 200 °C improves the compressive strength of linerboard by 10 to 15%. Plasma (atmospheric dielectric-barrier discharge) treatment is also reported to improve the wet tensile strength of linerboard (Johansson *et al*. 2006).

Several researchers are interested in the improvement of industrial grade papers with spent cooking liquor, also known as black liquor, from a chemical pulping process. Williston *et al*. (1967) suggested that the stiffness of linerboard could be improved by surface treatment (impregnation, dipping, or surface sizing) with waste liquor solids from chemical pulping. Mortarotti (1987) patented the idea of cardboard or semi-chemical paper treatment with black liquor to increase crush strength. The black liquor was added with at least 0.2% of polyvinyl alcohol. The three main natural polymers in wood are cellulose, hemicellulose, and lignin. Lignin concentration is high in the middle lamella followed by the primary wall, and lignin bonds to fibers in wood (*i.e*., lignin is a natural adhesive). In chemical pulping, lignin is degraded by chemicals such as NaOH and Na_2S and dissolved into the pulping liquors (Sjöström 1981). Black liquors contain dissolved lignin, carbohydrates such as hemicellulose and degraded cellulose, extractives, and spent cooking chemicals. Lignin and carbohydrates could work as natural adhesives.

Some researchers have tried to separate lignin from black liquor to utilize it in papermaking. Maximova *et al*. (2001) studied the effects of the addition of calcium ions and polyelectrolyte (poly-DADMAC) on the adsorption of kraft lignin precipitated from black liquor. Poly-DADMAC did not affect the adsorbed amount of lignin but did enhance the attachment strength of lignin on fiber surface, which resulted in increased paper strength. Maximova *et al*. (2004a) reported that dissolved lignin forms flocs and precipitates onto fiber surfaces with the addition of calcium and aluminum. Koljonen *et al*. (2004) examined the effect of pH on the formation of lignin precipitates, extractives, and metal ions on kraft pulp surfaces and reported that the strength properties of paper were slightly impaired by the precipitated materials. Antonsson (2007) precipitated black liquor and modified it with linseed oil to produce a hydrophobic lignin derivative, which was introduced to kraft liner to improve wet strength. He *et al*. (2014) reported that calcium, magnesium, and ferrum chlorides had significant effects on lignin solubility in kraft black liquor. Separating lignin from black liquor requires additional processes such as precipitation, which could increase the production cost for linerboard. The purification of lignin from the precipitated solids of black liquor would involve additional costs. This study used a simple approach utilizing black liquor, similar to Williston (1967) *et al*. In addition, the size of the materials in black liquor could influence the physical properties of paper, and a coagulant like aluminum ions could control the floc size of materials.

The objective of this study was to improve the strength of linerboard by the surface application of black liquor, *i.e*., spent cooking liquor from a kraft pulping process. The effect of the addition of aluminum sulfate (alum) in black liquor on the properties of linerboard were also examined. Alum was added to black liquor to coagulate solid materials in black liquor such as lignin and carbohydrates and to enlarge the size of the solid materials. Black liquors with various alum additions were applied on a linerboard by surface sizing with a laboratory inclined type surface size press. The effects of the addition level of alum in black liquor on properties of linerboard were evaluated in terms of the dry pick-up of black liquor.

EXPERIMENTAL

Materials

Corrugating medium was obtained from D paper mill in South Korea and used as a sample linerboard for the surface application of black liquor. The corrugating medium was produced from KOCC (Korean old corrugated container). The grammage of the corrugating medium was 140 g/m^2 and the thickness 225 μm. Thus, the bulk was 1.65 cm^3/g. The ash content was 19.5%.

Black liquor was received from M pulp mill in South Korea. The M pulp mill mainly produces HwBKP (hardwood bleached kraft pulp) from mixed hardwood chips. The solids concentration of the black liquor received was 39.2%, and the pH was 12.5. The ash content of the solids of the black liquor was 33%, and the lignin content was 24%.

Alum (aluminum sulfate, $Al_2(SO_3)_2 \cdot 14H_2O$) was used as coagulant and as a pH controller of black liquor. The Al_2O_3 equivalent was 17.16%.

Methods

Surface sizing

The linerboard was cut to 20 cm × 20 cm, conditioned overnight at 23 ± 1 °C and 50 ± 2% RH (relative humidity), and placed into zip-lock bags before surface sizing. A laboratory inclined, pond sized press (GIST, Daejeon, Republic of Korea) was used to apply black liquor. The velocity (3 m/min), nip pressure (6 kg_f/cm^2), and size solution temperature (30 °C) were kept constant for all experiments. To vary dry pick-ups, the solid contents of black liquor were adjusted from 5% to 20% (w/w) by adding distilled water. Alum solution (10% concentration, w/w) was added to the black liquor solution to regulate the pH of the black liquor. The added amount of alum was 0%, 2.5%, 12.3%, and 24.5%, based on oven dry weight of black liquor solids. The pH of black liquor varied according to the added amount of alum, which ranged between 12.3 (0% alum), 11.7 (2.5% alum), 10.3 (12.3% alum), and 7.6 (24.5% alum). The viscosity of black liquor was measured with a Brookfield viscometer (DV-II pro, spindle no. 61, Middleboro, USA) at room temperature (18 °C) and at 100 rpm.

A sample of corrugated medium was taken from the zip-lock bags for surface sizing. Sized papers were dried with a laboratory drum drier at 100 °C (DaeIl Mechanics, Daejeon, Republic of Korea). The papers were conditioned overnight at 23 ± 1 °C and 50 ± 2% RH. The dry pick-up (g/m^2) of black liquor solids was calculated after drying and conditioning,

$$\text{Pick}-\text{up}\ [\frac{\text{g}}{m^2}] = M_s - M_b \tag{1}$$

where M_s represents the grammage of the sized paper and M_b is the grammage of the base paper.

Paper analysis

The surface-sized sheets were conditioned overnight at 23 ± 1 °C and 50 ± 2% RH before analysis. Tensile strength and tensile energy absorption (TEA) were measured with a horizontal tensile tester (L&W, Kista, Sweden) according to ISO 1924-3 (2005). The compressive strength was evaluated with the Ring Crush Tester (RCT, L&W, Kista, Sweden) according to ISO 12192 (2011). Bursting strength was measured according to ISO 2758 (2001) with a Mullen bursting strength tester (L&W). Folding endurance was tested with an MIT folding endurance tester (Tinius Olsen, Horsham, USA) according to ISO 5626 (1993). Air permeability was quantified with the Gurley tester (FRANK-PTI, Birkenau, Germany; ISO 5636-5 (2003)). To evaluate hydrophobicity, the Cobb test (exposure time to water: 10 sec; ISO 535 (1991)) and contact angle measurements were performed (Pocket goniometer, Testing Machines, Inc., New Castle, USA; ASTM D724-99 (2003)).

RESULTS AND DISCUSSION

Effect of Alum on Black Liquor Viscosity

The original pH of black liquor was 12.5. Alum was added to the black liquor to regulate its pH and coagulate solids. The amount of alum added was increased to 24.5% on black liquor solids to reduce the pH to 7.6. However, regulating the concentration of black liquor affected the pick-up of black liquor solids. The black liquor concentration and the degree of coagulation of solid materials in it could influence the viscosity of sizing liquor (black liquor), which is an important factor in the surface sizing process.

Figure 1 shows the effect of black liquor concentration and the alum dosage on the Brookfield viscosity of black liquors. For black liquor alone or with 2.45% alum addition, an increase in the concentration of black liquor slightly increased the viscosity. When the concentration of black liquor was increased from 5% to 20%, the viscosity of black liquor increased from 3 cPs to 8 cPs. When the black liquor was at 10.8 cPs, there was a 2.45% alum addition, and the solid content was 20%. For the 12.3% alum addition, the viscosity steeply increased after 10% black liquor concentration. At 20% black liquor concentration and alum addition of 12.5%, the viscosity of black liquor increased to 115.5 cPs. With alum addition of 24.5%, it decreased to 94.5 cPs. These results show that the variation in the viscosity of black liquor with increasing black liquor concentration was not noticeable at a low dosage of alum, while the viscosity increased exponentially at higher dosage. These effects could influence the runnability of size press and penetration of sizing liquor into paper, affecting strength properties of paper.

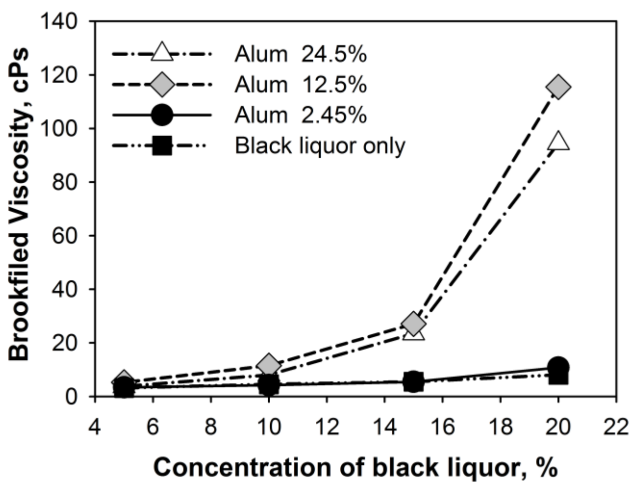


Fig. 1. The effect of black liquor concentration and the added amount of alum on black liquor viscosity

The increased viscosity of black liquor indicated that solids in black liquor were coagulated with alum. Variations in the particle size of solids in black liquor were not measured in this study. However, it has been reported that low molecular weight lignin is agglomerated with a coagulant such as aluminum or metal ions (Yaser *et al*. 2014). According to Richardson (1988), the mean particle size of kraft lignin from pulp mills was in the range of 8.6 to 26.1 μm. Öhman and Theliander (2007) reported that the particle size of precipitated lignin was smaller at a higher pH and lower temperature, and the particle size peaked from 14 μm to 45 μm.

Effects on Strength Properties of Corrugated Medium

A noticeable increase in the tensile strength of linerboard in the machine direction (MD) and cross direction (CD) was not observed with the surface sizing of black liquor (Fig. 2, Black liquor only). However, noticeable improvement in the tensile index was shown with 2.5% alum (Fig. 2). In MD, tensile strength linearly increased until 3.6 g/m^2 of dry pick-up of black liquor solids. In CD, tensile strength linearly increased with dry pick-up. In MD, the tensile index was increased by 23.2% from 44.31 Nm/g to 54.90 Nm/g and was increased 26.2% from 16.25 Nm/g to 20.50 Nm/g in CD. When alum was added to black liquor at dosages of 12.3% and 24.5%, the tensile strength was still higher than that of black liquor only; however, they were much lower than the 2.5% alum addition. Similar tensile strength values were observed in the 12.3% and 24.5% alum additions.

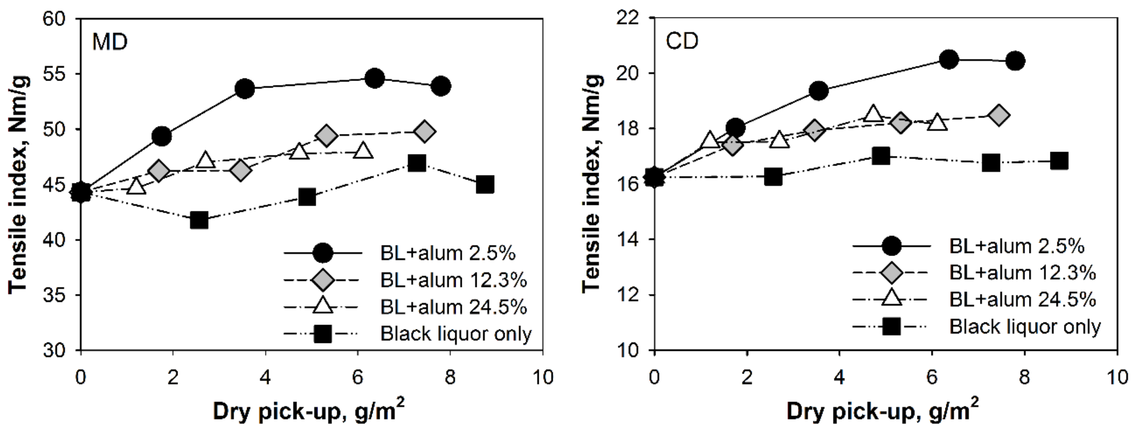


Fig. 2. The effect of dry pick-up of black liquor on tensile strength in MD (left) and in CD (right) of corrugated medium at various addition levels of alum

The tensile energy absorption (TEA) is proportional to the work required to break a paper strip and is influenced by the elongation and stress of a paper. It is an important property for packaging paper like sack kraft. The paper must be strong enough to resist tough handling and also absorb the shock during handling (Meinander 2000).

The corrugated medium surface-sized with only black liquor showed the highest TEA in both MD and CD (Fig. 3).

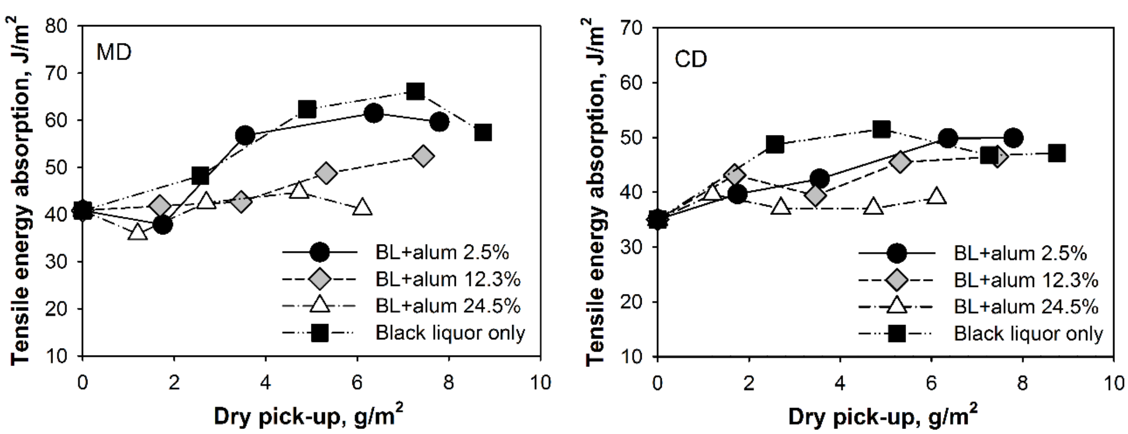


Fig. 3. The effect of dry pick-up of black liquor on TEA (tensile energy absorption) in MD (left) and in CD (right) of corrugated medium at various addition levels of alum

TEA in MD increased 61.9% at the dry pick-up of 7.27 g/m^2, while that in CD increased 47.2% at the pick-up of 4.9 g/m^2. Unlike TEA, the tensile strength of the corrugated medium treated with black liquor was the lowest (Fig. 2). This result suggests that surface sizing with black liquor remarkably improved the elongation of the paper.

When 2.5% alum on black liquor solids was added, the surface-sized linerboard showed excellent TEA (Fig. 3; BL+alum 2.5%), which was similar to the linerboard treated with black liquor (Fig. 3; Black liquor only). At the dry pick-up of 6.36 g/m^2, TEA in MD increased 50.5% and 42.4% in CD. However, higher alum addition levels deteriorated TEA. When 12.3% or 24.5% alum was added, TEAs decreased with increasing alum addition at any given dry pick-up.

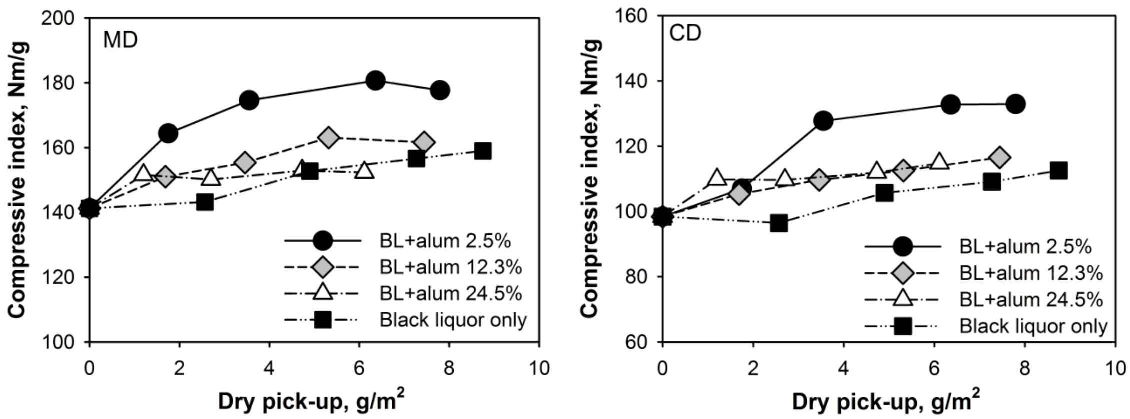


Fig. 4. The effect of dry pick-up of black liquor on compressive strength in MD (left) and in CD (right) of corrugated medium at various addition levels of alum

Compressive strength is the most important physical property of linerboard, especially for corrugated medium. Compressive strength was evaluated with ring crush strength. Compressive index showed similar trends to tensile strength (Fig. 4). Increasing the dry pick-up of black liquor improved the compressive index, which confirmed that solid materials in black liquor increased bonds between fibers and improved paper strength. The lowest compressive index was observed when the corrugated medium was treated with only black liquor (Fig. 4; Black liquor only). When the dry pick-up was 8.75 g/m^2, the compressive index in MD and CD improved to 12.5% and 14.4%, respectively. A slight addition of alum further increased the compressive strength. The highest increase in compressive index was observed with 2.5% alum (Fig. 4; BL+alum 2.5%). When the dry pick-up was 6.35 g/m^2, the compressive strength in MD and CD was improved by 27.9% and 34.9%, respectively, compared with the control value. However, further addition of alum slightly impaired the compressive strength. When the alum addition was increased to 12.3% and 24.5%, the compressive index decreased compared with the 2.5% alum addition at similar dry pick-ups. However, the value was still higher than the linerboard surface-sized with only black liquor. This result indicated that surface sizing with black liquor and the optimum amount of alum effectively improved compressive strength.

Bursting strength linearly increased with an increase in the dry pick-up of black liquor when the liner was surface-treated with black liquor only (Fig. 5; Black liquor only). It was increased 17.5% at the dry pick-up of 4.9 g/m^2 and 21.4% at 8.75 g/m^2. When 2.5% alum was added to the black liquor (Fig. 5; BL+alum 2.5%), the burst index showed slightly higher values than for black liquor only. At the dry pick-up of 7.8 g/m^2, the burst index increased 25.4%. With 12.3% alum addition, the burst index showed similar values to the case treated with black liquor only. The lowest bursting strength was observed when 24.5% alum was added. At the pick-up of 6.11 g/m^2, the burst index was increased 8.0%.

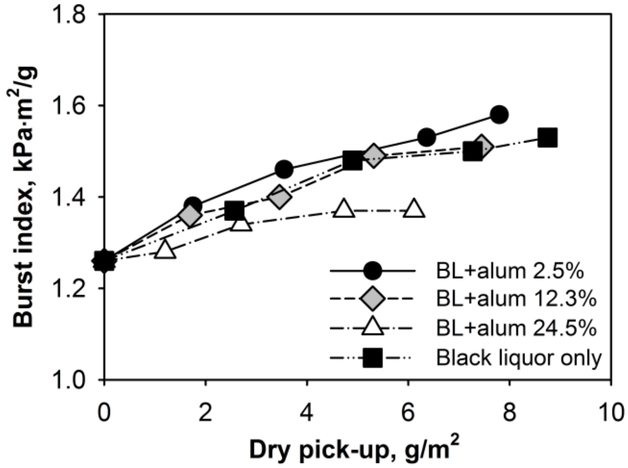


Fig. 5. The effect of dry pick-up of black liquor on bursting strength of corrugated medium at various alum levels

These results implied that surface sizing with black liquor effectively improved the strength properties of linerboard including tensile strength, TEA, compressive strength, and bursting strength. A small addition of alum (2.5% on black liquor solids) promoted the effect of surface sizing. The surface application of black liquor with alum could be applied to other industrial grade papers such as sack kraft paper and test liner, which need high tensile strength, TEA, and bursting strength. To improve paper strength, sizing liquor and the solids in the liquor should penetrate into the pores of the paper and increase the bonds between fibers. During chemical pulping, lignin and carbohydrates are degraded by pulping chemicals and dissolved into pulping liquors (Sjöström 1981). The dissolved lignin and carbohydrates in black liquor could work as bonding agents between fibers. Black liquor was very effective in increasing TEA and bursting strength. The aggregated wood components with 2.5% alum addition, *i.e.*, the enlarged solids such as lignin and carbohydrates, were more efficient in increasing bonding between fibers.

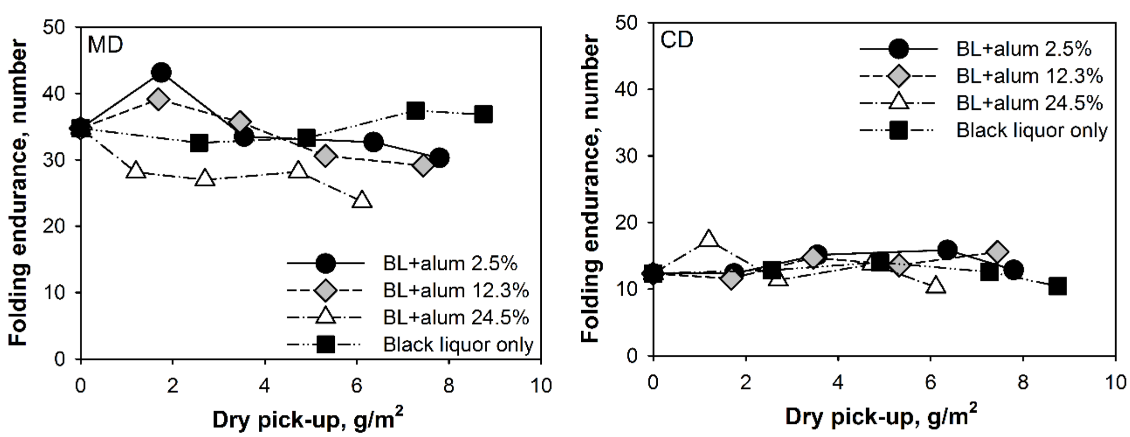


Fig. 6. The effect of dry pick-up of black liquor solids on folding endurance in MD (left) and in CD (right) of corrugated medium at various addition levels of alum

Alum could interact with the negatively charged groups within the black liquor solids and coagulate the negatively charged solids, enlarging the solids. In addition, alum could improve adsorption of the negatively charged solids within the black liquor on fiber surface, increasing interactions between the negatively charged solids and fiber surface. However, the excessive addition of alum caused adverse effects on paper strength. The possible explanations are that the enlarged aggregates of dissolved lignin and carbohydrates with increased viscosity could not penetrate into pores between fibers in paper and/or that the excessive aggregation could influence adverse effects on the film-forming ability of materials in black liquor.

Unlike other strength properties, folding strength was not noticeably influenced by the surface sizing of black liquor with or without the addition of alum (Fig. 6). When the corrugated medium was surface-sized with black liquor only, the folding strength was increased 7.7% in MD and 1.9% in CD at the pick-up of 7.27 g/m^2. When 2.5% alum was added, the folding strength was increased about 25% at the pick-up of 1.75 g/m^2. Then, the folding strength slightly decreased, which resulted in a lower strength than the control. In CD, the number of double folds was increased from 12.3 to 15.9 (28.6% increase). The excessive addition of alum deteriorated folding strength. When 12.3% of alum was added and the pick-up was 7.44 g/m^2, the number of double folds was increased from 12.3 to 15.6 (26.3% increase) in CD and decreased from 34.8 to 29.2 (16.1% decrease) in MD. When 24.5% of alum was added, the number of double folds decreased 31.8% in MD and 16.6% in CD at the pick-up of 6.11 g/m^2.

Effect on Structure and Surface Properties of Corrugated Medium

Increasing dry pick-up increased air permeability (Fig. 7). Air permeability was evaluated with the Gurley tester, which measured the time required for 100 mL of air to flow through a paper sample. The increased air permeability showed that solids from black liquor penetrated into the pores of the paper and filled the voids between fibers or blocked the pores on the paper surface. The air permeability did not vary noticeably when the dry pick-up was increased, until 5 g/m^2. After 6 g/m^2, it started to increase. With 2.45% alum, the air permeability was the highest at a given dry pick-up, which revealed that the alum affected floc size or film-form-ability of materials in black liquor.

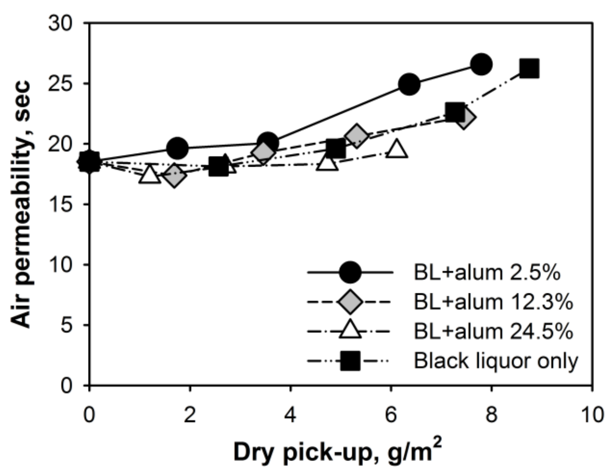
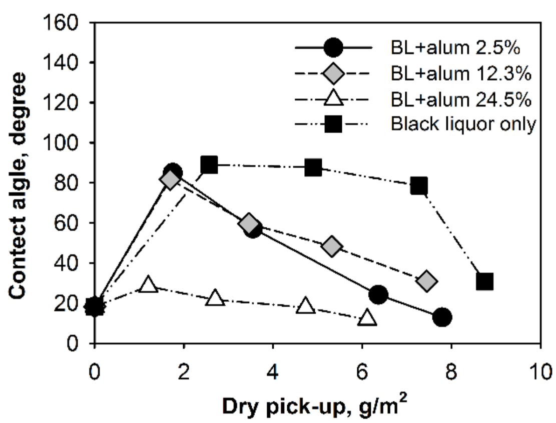


Fig. 7. The effect of dry pick-up of black liquor on air permeability of corrugated medium at various alum levels

At the dry pick-up of 7.8 g/m^2, air permeability increased 43.4% from 18.5 sec to 26.6 sec. The 24.5% alum addition showed the lowest air permeability. At the pick-up of 6.1 g/m^2, air permeability increased 4.5% only. This might be proof of the poor film-forming ability of excessively coagulated materials in black liquor. It seemed that the 2.5% addition of alum coagulated low molecular weight and dissolved lignin and carbohydrates. It also appears to have helped the film-forming, blocking, or filling of voids in paper. However, excessive addition of alum seemed to deteriorate the film-forming ability of solids in black liquor.

Figure 8 shows the effect of the dry pick-up on sizing degree or hydrophobicity of the corrugated medium at various levels of alum. Without the surface sizing of black liquor, the contact angle of water on the corrugated medium was around 20°, which meant that the tested sample was hydrophilic. Surface sizing of black liquor increased the hydrophobicity of the corrugated medium (Fig. 8; Black liquor only). The contact angle initially increased with dry pick-up. Interestingly, after reaching a maximum at around 2.6 g/m^2 of dry pick-up, the contact angle slightly decreased with increasing pick-up. At 8.75 g/m^2 the contact angle was decreased to the value of unsized corrugated medium. The Cobb test showed similar results with the contact angle (Fig. 8). The lowest Cobb value was observed at around 2.6 g/m^2 of dry pick-up when the paper was treated with black liquor only. After that, the Cobb value was increased to a similar value as the non-treated sample and consequently became hydrophilic. The corrugated medium became hydrophobic at a lower pick-up, while it was hydrophilic at higher pick-up. The reason for this phenomenon is unknown. Kraft lignin is a hydrophobic material. The lignin adsorbed on pulp fiber and mica surface increases the contact angle of water (Maximova *et al.* 2004b). As more lignin adsorbs onto the surface, hydrophobicity increases. In contrast, carbohydrates are hydrophilic. When more carbohydrates are exposed on the outer layer of fibers after surface sizing, the paper is more likely to be hydrophilic. The changes in hydrophobicity could be related to the chemical compositions in the outer layer of the surface sizing film. This phenomenon needs further investigation.



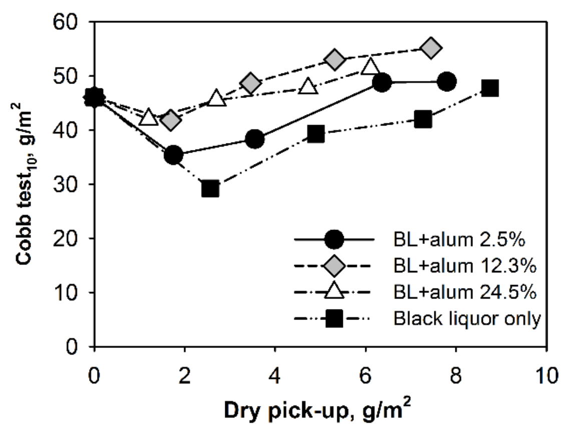


Fig. 8. The effect of dry pick-up of black liquor on contact angle of water (left) and Cobb test (right) of corrugated medium at various addition levels of alum

The addition of alum also influenced the hydrophobicity of paper. Increasing the amount of alum decreased the contact angle of water and increased the Cobb values at a given dry pick-up. The addition of alum made the corrugated medium hydrophilic. The structure of the lignin adsorbed layer also influenced hydrophobicity (Maximova *et al.*

2004b). It has been reported that maximum hydrophobicity is obtained when the adsorbed lignin layer forms a granular structure. When cationic polymer is added, it forms complexes with lignin and affects the contact angle. At a higher alum addition, alum could form complexes of solids within black liquor, which might hinder the orientation of hydrophobic materials on fiber surface. In addition, the excess addition of alum could influence the film-forming ability of black liquor, which could affect penetration of water. These could increase hydrophilicity of the surface sized paper. This effect also needs further investigation.

Surface pictures of the base paper and the surface sized samples with relatively higher pick-ups are shown in Fig. 9. No noticeable difference was observed among the base paper (Fig. 9a), the corrugated medium treated with black liquor only (Fig. 9b), and the sample treated with black liquor and 2.5% alum (Fig. 9c). A noticeable amount of the black liquor solids, *i.e.*, lignin and carbohydrates, was not observed on the surface of the corrugated medium. Thus, most of the black liquor solids that were applied with surface sizing penetrated the pores of the paper, which supported the hypothesis that lignin and carbohydrates in black liquor penetrate paper pores and increase the bonds between fibers in the paper.

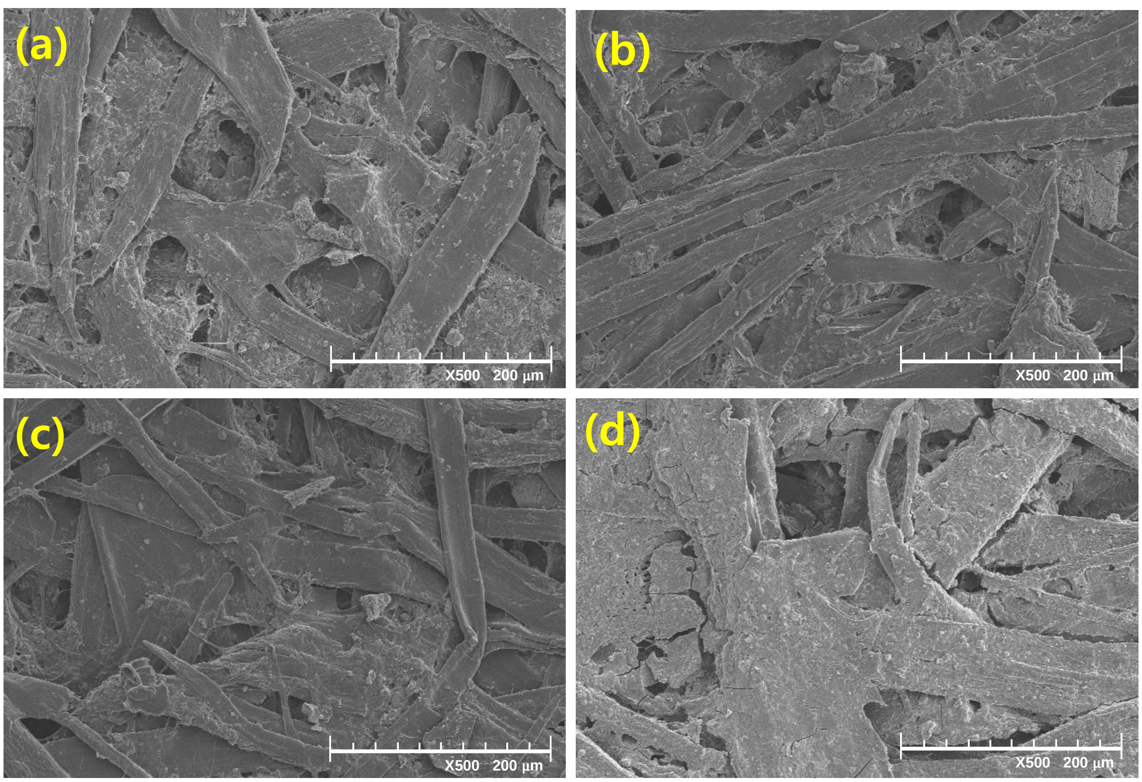


Fig. 9. SEM images of corrugated medium surface: (a) base paper, (b) surface sized with only black liquor (dry pick-up: 7.27 g/m^2), (c) surface sized with black liquor and 2.5% alum (dry pick up: 7.79 g/m^2), (d) surface sized with black liquor and 24.5% alum (dry pick-up: 6.11 g/m^2)

However, a layer of solids was observed on the surface of the corrugated medium treated with black liquor and 24.5% alum. In addition, many cracks were observed in the surface sizing layer. This showed that an excessive addition of alum formed larger aggregates of lignin and carbohydrates, which were too large to penetrate the paper pores.

Also, an excessive addition of alum deteriorated the film-forming ability of black liquor solids by excessive coagulation of solid particles. This might be the one reason that the strength properties deteriorated with a higher alum addition.

CONCLUSIONS

1. Surface sizing of black liquor efficiently improved the dry strength of corrugated medium. There was a distinctive increase in TEA and bursting strength with the surface application of black liquor, while folding endurance was increased in limited conditions.
2. The addition of alum into black liquor increased the strength properties of corrugated medium. An addition of 2.5% alum showed the highest increase in tensile strength, TEA, compressive strength, and bursting strength. Further additions of alum deteriorated strength properties.
3. Air permeability slightly increased in the range of dry pick-up of 3 to 5 g/m^2, while it noticeably increased after 5 g/m^2.
4. Black liquor penetrated the paper pores. Materials such as lignin and carbohydrates in black liquor increased bonds between fibers, which resulted in improved dry strength. There is an optimum floc size of black liquor solids for strength improvement.
5. The surface sizing of black liquor made the corrugated medium hydrophobic at a low pick-up (around 2 to 3 g/m^2). After the maximum hydrophobicity, the hydrophilicity increased with increasing dry pick-up. Thus, the contact angle decreased and Cobb size increased. In addition, the hydrophilicity of the corrugated medium increased with the added amount of alum.

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