A Filler Distribution Factor and its Relationship with the Critical Properties of Mineral-Filled Paper

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The use of mineral fillers in the paper industry has attracted much attention due to its low cost and ability to improve optical properties and printability. Besides the filler characteristics, paper properties, such as bulk, tensile, and opacity, are greatly affected by filler distribution in the z-direction. Therefore, optimization of filler distribution is an effective way to maximize the value of fillers. In this work, a filler distribution factor (F_c) was proposed to quantitatively describe the concentrated degree of filler distribution in the z-direction. The reduction in F_c resulted in an increase in paper bulk, porosity, and opacity, due to the generation of more interfaces between fibers and fillers. When filler particles were concentrated in one layer (F_c = 1), the tensile strength of the filled paper increased between 26 to 40% in comparison to the paper with various F_c values. For a given F_c, better tensile and opacity properties were achieved by increasing filler concentration on the surface layer of paper.

Keywords: Mineral fillers; Filler distribution; Paper strength; Optical properties

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INTRODUCTION

Mineral fillers have been widely used in the paper industry because of their advantages, including improvement of paper properties and energy and cost savings. Besides filler type and characteristics (Han and Seo 1997; Thorn and Au 2009; Chen *et al*. 2011; Hubbe and Gill 2016; Fan *et al.* 2017), filler distribution in the cross-section of paper can directly affect many end-use properties of paper products, such as strength, brightness, opacity, and ink receptivity (Modgi *et al.* 2004; Li *et al.* 2017). Therefore, the requirements of filler distribution in paper varies greatly according to the paper quality and grades.

For copy paper, dusting can be mitigated when the filler content is lower on the paper surface than in the center (Ahlroos *et al*. 1998; Gron and Ahlroos 1998). For coated paper and super-calendered paper grades, the filler content on the paper surface should be higher (Odell 2000). Increased filler content on the paper surface can improve paper brightness and decrease the bending stiffness and surface roughness. In addition, paper strength can be enhanced *via* increasing filler concentration on the paper surface (Puurtinen 2003).

Filler distribution can be affected by dewatering conditions, which influences the movement of fillers and fines in the cross-section of paper. Therefore, the type of paper machine, dewatering rate, suction boxes, and paper basis weight can be the factors governing filler distribution. It has been shown that sheet preparation on a Fourdrinier paper machine leads to a low filler content on the wire side due to the removal of filler particles induced by suction boxes from the wire side. Hybrid formers tend to reduce the filler content also in the top side, resulting in a more symmetrical filler distribution in the cross-sectional area of the paper. In the case of a gap former, water is drained simultaneously from both sides of the paper, which provides a symmetrical distribution with a higher filler content at the center of the paper.

The development of the multilayer headbox and multi-layering forming provides more possibilities for the design of the structure of paper and paperboard. The filler content can be altered in each layer, thus, improving paper properties. To investigate filler distribution in the z-direction, Puurtinen (2004) proposed two factors to characterize filler distribution, *i.e.*, symmetry factor (F_{sym}) and shape factor (F_s), which are defined as:

$$F_{sym} = (F_t - F_b) / min(F_t, F_b) \quad (1)$$

$$F_s = (F_t + F_b - 2F_m) / min(2F_m, F_t + F_b) \quad (2)$$

where F_t, F_m, and F_b are the filler contents (%) in the top, middle, and bottom layer (near forming wire) of laminated paper, respectively.

The symmetry factor (F_{sym}) reflects the differences in filler content on both sides of the paper. Shape factor (F_s) represents the linearity or nonlinearity of the curve of the filler distribution. Both factors can describe the distribution curve well. It was found that asymmetrical filler distributions led to a lower porosity, and symmetrical filler distributions contributed to oil absorption of two sidedness (Puurtinen 2004). However, the effects of filler distribution on paper strength and optical properties were not investigated. These equations may not be suitable for laminated paper whose filler content is zero in one of the layers.

In this study, a new filler distribution factor (F_c) was proposed to describe how filler particles are concentrated in the z-direction of laminated paper, and the feasibilities of improving paper strength and optical properties by adjusting F_c were investigated. Since the multilayer headbox and multi-layering forming have been commercialized, this finding can be potentially applied to writing/printing paper and paperboard products.

EXPERIMENTAL

Materials

The pulp fibers used for the formation of handsheets were commercially bleached hardwood kraft pulp board with a brightness of 84.4%ISO. The pulp was refined to a freeness of 405 mL by PFI mill. The filler, a fly ash based calcium silicate (FACS) with average particle size of 21 μm and brightness of 84.4%ISO, was supplied by a coal-fired power (thermal power) plant in China and was mainly composed of calcium silicate. Compared to PCC or GCC, FACS can improve paper bulk remarkably which can be potentially used in light-weight paper (Song *et al*. 2012). High molecular weight cationic polyacrylamide (CPAM) Percol 182 was provided by BASF (Nanjing, China).

Methods

Handsheet preparation and testing

The hardwood pulp was disintegrated at a consistency of 1.2 wt% and diluted with water to a consistency of 0.3 wt%. Filler slurries were prepared at 10 wt% solid content and subsequently added to the pulp slurries. The retention aid (CPAM) was added into the fiber furnish at a dosage of 0.03 wt% based on the oven-dried pulp amount.

Because of the difficulty in controlling filler distribution accurately in handsheets, the handsheets with various filler distribution were controlled by laminating three layers of the handsheet at 40 g/m^2 (*i.e.*, 0.80 g) after forming. To eliminate the variation of paper properties induced by the differences in filler content, the handsheets for each layer with target filler content were made according to filler retention rate and ash testing results; then the damp plies were combined as layered sheets before pressing and drying. The total filler content in the laminated paper was fixed at 25%±0.5%. Seven filler distribution curves in the z-direction of paper were designed by adjusting the filler content in three layers of the handsheet, as shown in Table 1.

The handsheets were made according to TAPPI standard method T 205 sp-95 (2002). The wet laminated papers were pressed, and then air-dried at 23 °C and 50% relative humidity for 24 h before physical testing. At least five series of experiments were run for each formulation. The bulk (T411om-89, 2005), Gurley porosity (T460 om-02, 2002), tensile index (T494 om-01, 2001), internal bond strength (T541 om-05, 2005), brightness (T452 om-02, 2002), and opacity (T425 om-01, 2001) were measured according to TAPPI test methods. The handsheets were calcined at 525 °C to determine the filler content in handsheets according to TAPPI test method T211 om-93 (2002).

Table 1. Design of Filler and Fiber Mass Distribution in Each Layer

Sample No.	Top Layer		Middle Layer		Bottom Layer	
	Fiber (g)	filler (g)	fiber (g)	filler (g)	fiber (g)	filler (g)
1*	1.8	0.6				
2	0.8	0	0.2	0.6	0.8	0
3	0.5	0.3	0.6	0.2	0.7	0.1
4	0.6	0.2	0.6	0.2	0.6	0.2
5	0.4	0.4	0.7	0.1	0.7	0.1
6	0.5	0.3	0.7	0.1	0.6	0.2
7	0.6	0.2	0.5	0.3	0.7	0.1
8	0.7	0.1	0.4	0.4	0.7	0.1

*The sheet sample from No.1 is once-formed sample.

Scanning electron microscopy observation

The cross sections of the paper samples were characterized with a JEOL JSM-6400 scanning electron microscope (Quebec, Canada). The back-scattered electron images (BEI) were used to observe filler distribution in the z-direction with an accelerating voltage of 15 kV. Samples were carbon-coated using an Edwards E306A evaporative golden coater (Quebec, Canada) before analysis.

Filler distribution factor

Filler particles typically exist in paper in the form of aggregates due to the flocculation by using chemical additives. The formation of filler aggregates indicates a local rich filler concentration, which impacts paper structure, and thus affects the paper strength and optical properties.

Herein, a new filler distribution factor, F_c (Eq. 3), was proposed to describe the degree of filler particles concentrated in the z-direction of paper for a given filler content. The F_c of different samples in Table 1 is shown in Table 2.

$$F_c = [max\ (F_t, F_m, F_b) - min\ (F_t, F_m, F_b)]/\ max\ (F_t, F_m, F_b) \quad (3)$$

where F_t, F_m, and F_b are the filler content (g) in the top, middle, and bottom layer (near forming wire) of laminated paper, respectively.

Table 2. Filler Distribution Factor (Fc) of Different Paper Samples.

Sample No.	2	3	4	5	6	7	8
Fc	1.00	0.67	0	0.75	0.67	0.67	0.75

A higher value of F_c indicates that more filler particles are concentrated in one layer of the laminated paper. $F_c = 1$ implies that no filler is present in at least one layer of the laminated paper, while $F_c = 0$ implies that the filler is distributed evenly among the layers.

RESULTS AND DISCUSSION

Filler Distribution in Cross-Section of Paper

Figure 1 illustrates the filler distribution in the cross-section of paper. The filler distribution in the cross-section was varied based on the design in Table 1. It is noted that the image of sample 2, in which all filler particles concentrated in the middle layer of the handsheet, could not be obtained because the inter-bonding strength of the sample was too weak, such that the structure was destroyed easily during the sample cutting for SEM observation. A comparison with sample 0 (without filler) shows that the thickness of the paper was increased noticeably when FACS filler was employed, which was also verified in previous studies (Song *et al.* 2012; Zhang *et al.* 2013). In addition, the differences in filler distribution resulted in the variation of paper thickness.

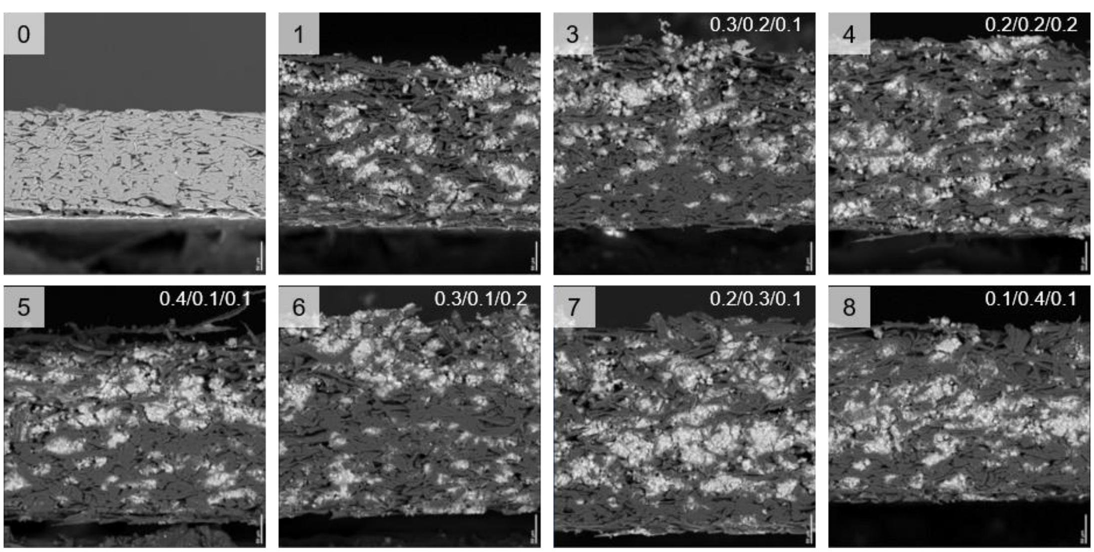

Fig. 1. SEM images of cross-section of laminated paper. The scale bar represents 50 μm.

Bulk and Porosity

The effect of F_c on paper bulk is presented in Fig. 2A. As shown, the bulk of the filled paper varied with F_c while the filler content in the paper was fixed at around 25%. Compared to the paper without a laminated formation, the bulk of laminated paper was increased at different levels by optimizing F_c. A higher F_c resulted in lower bulk. When filler particles were highly concentrated in one layer, *i.e.*, sample 2 ($F_c = 1$), the paper bulk decreased by 11.2% compared to the sheets with even filler distribution, *i.e.*, sample 4 ($F_c = 0$). In addition, the sheets with the same F_c exhibited similar bulk. Uniform filler distribution in the cross-section helped to maximize the paper bulk. Filler particles concentrated in one layer can readily form large aggregated particles and thus, enhance its packing ability and decrease the interfaces between fiber and filler. As a result, paper bulk was decreased evidently.

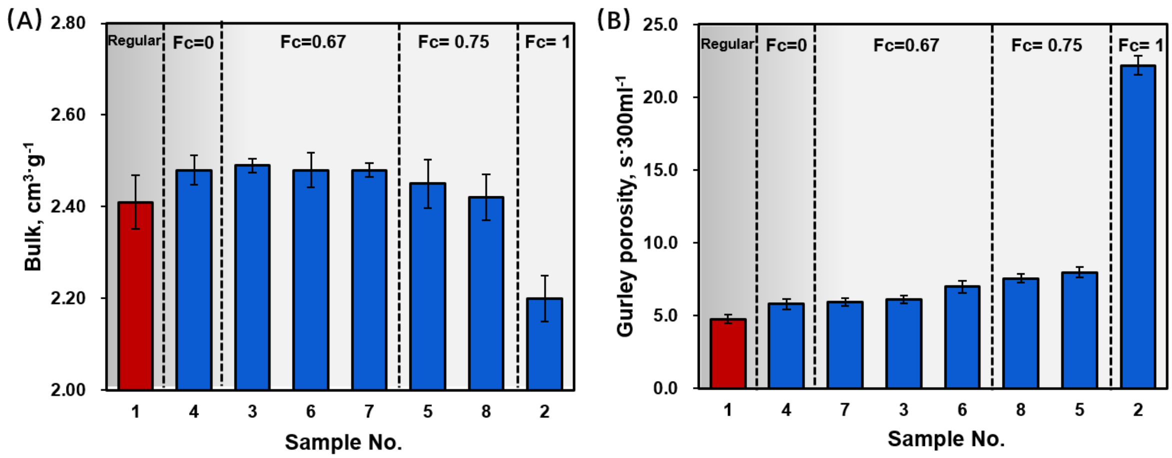

Fig. 2. Effect of filler distribution factor on paper bulk (A) and porosity (B)

For a given filler content, bulk increase can be ascribed to by the pores induced by filler addition to the cellulosic fiber matrix (Brown 1998; Hubbe and Gill 2004). However, this would also compromise the air resistance of the filled paper. As shown in Fig. 2B, the air resistance of FACS filled paper increased as the filler particles were concentrated in one layer. It is also noted that, for a given F_c, decreasing the filler content in the middle layer tended to increase the air resistance of the filled sheets. In general, paper with a higher bulk typically led to a lower air resistance due to having more pores in the paper. Compared to the paper with even filler distribution, the laminated paper with a higher F_c indicated the filler content in some layers was lower, which was conducive to form more fiber-fiber bonds and decreased the number of pores. In addition, filler particles concentrated in one layer also increased the packing ability of particles, resulting in an increased air resistance.

Strength Properties

Figure 3A shows the effect of F_c on the tensile index of FACS filled paper. As shown, the laminated paper had a higher tensile index compared to once formed paper, and the tensile index can be improved by optimizing F_c. When $F_c = 1$, the paper strength increased between 26 to 40% compared to that of other samples, indicating that the fiber-fiber bonding can be varied with filler distribution. Additionally, Fig. 1 and 3A indicated that increasing the filler content in the surface layer, *i.e.*, top or bottom layer, helped to increase paper tensile index for a given F_c, which was also reported in the literature (Puurtinen 2003).

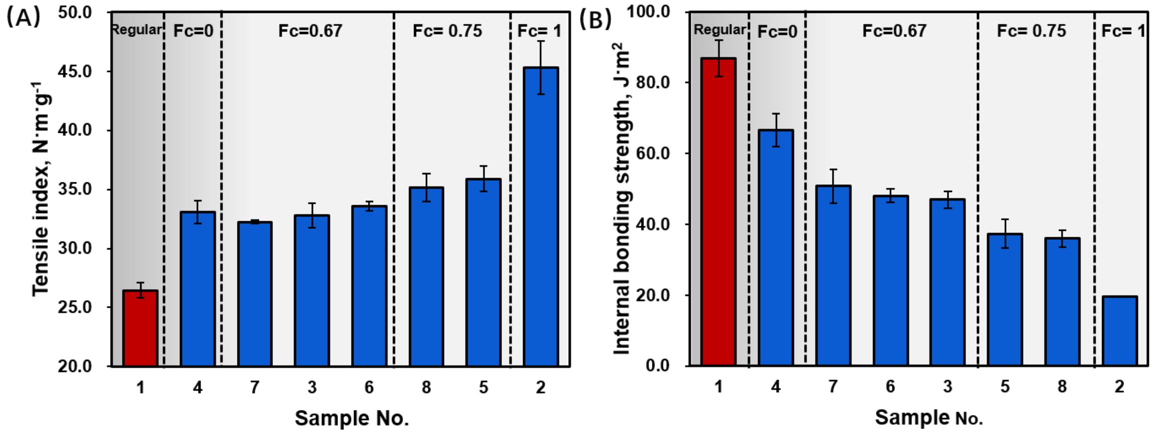

Fig. 3. Effect of filler distribution factor on paper tensile index (A) and internal bonding strength (B)

The use of mineral filler generally reduces paper tensile index (Huang *et al.* 2014; Hubbe and Gill 2016; Ting *et al.* 2016). The tensile index of the paper largely depends on inter-fiber bonding (Hubbe 2014). The decrease in tensile index was partly linked to the substitution of cellulosic fiber by a mineral filler for a given paper grammage. Importantly, filler particles hindered the formation of hydrogen bonding between cellulosic fibers. In this work, the effect of filler on paper tensile strength was linked to filler distribution. As F_c increased, more filler particles tended to be rich in one layer of the paper and more filler aggregates would be formed, which helped to enhance the packing ability of particles and enhanced fiber bonding. This resulted in the improvement of the strength of the laminated paper. In the paper industry, the use of filler with board particle size distribution (Velho 2002) such as GCC, or application of pre-flocculation of fillers (Peng *et al.* 2015), can also enhance the packing ability of fillers and endow the paper with better strength. In contrast, when filler distribution became uniform in the z-direction, the paper strength could be affected from two aspects. On one hand, more particles prevented fiber bonding, and thus, reduced paper strength. On the other hand, the improvement in paper uniformity was conducive to reduce the local stress concentration when tensile stress was applied to the paper. This could explain the similar tensile index of paper with F_c = 0 and F_c = 0.67. It was also noted that a subtle variation in tensile strength between the sheets for a given F_c. It is also reported that the paper strength increased with increased filler concentration of the paper surface (Puurtinen 2003). The higher filler content in the top or bottom layer typically caused a higher filler content on the surface of the paper, which can alleviate the negative effect of filler particles on paper strength.

Differing from tensile index, the inter-bonding strength of the filled paper decreased as F_c was increased, as illustrated in Fig. 3B. A highly concentrated filler distribution in the middle layer resulted in the weakest inter-bonding strength. For laminated sheets, inter-bonding mainly depends on the fiber-fiber bonding and the bonding strength between sheet layers. Increasing the filler concentration in one layer not only prevents fiber-fiber bonds but also decreases the bonding between the sheet layers. This effect would be pronounced when the filler was highly distributed in the middle layer, such as sample 2.

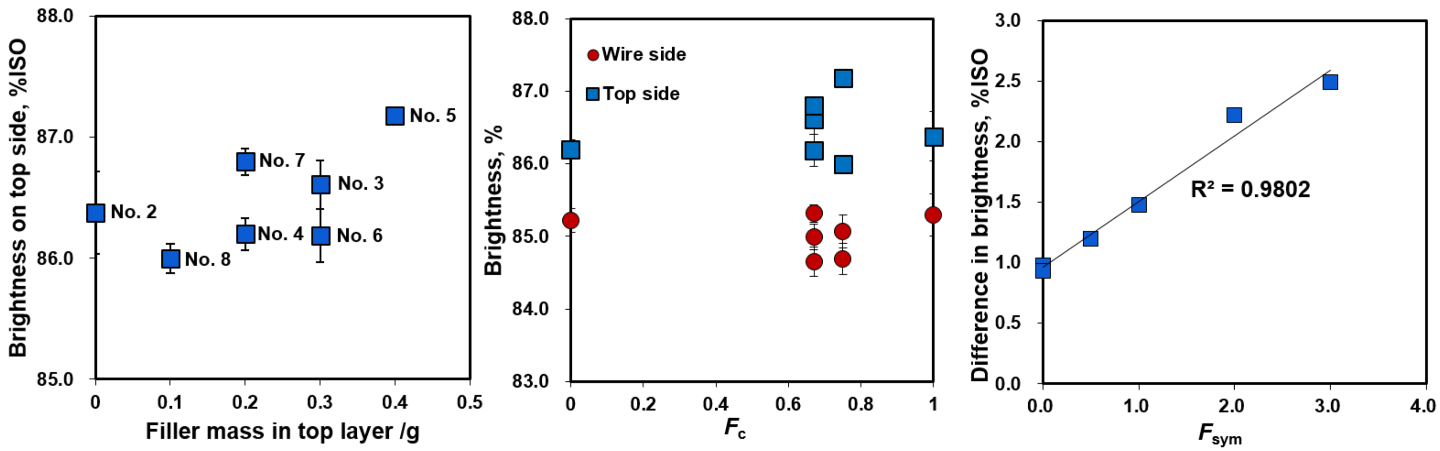

Fig. 4. Effect of filler distribution in Z direction on paper brightness

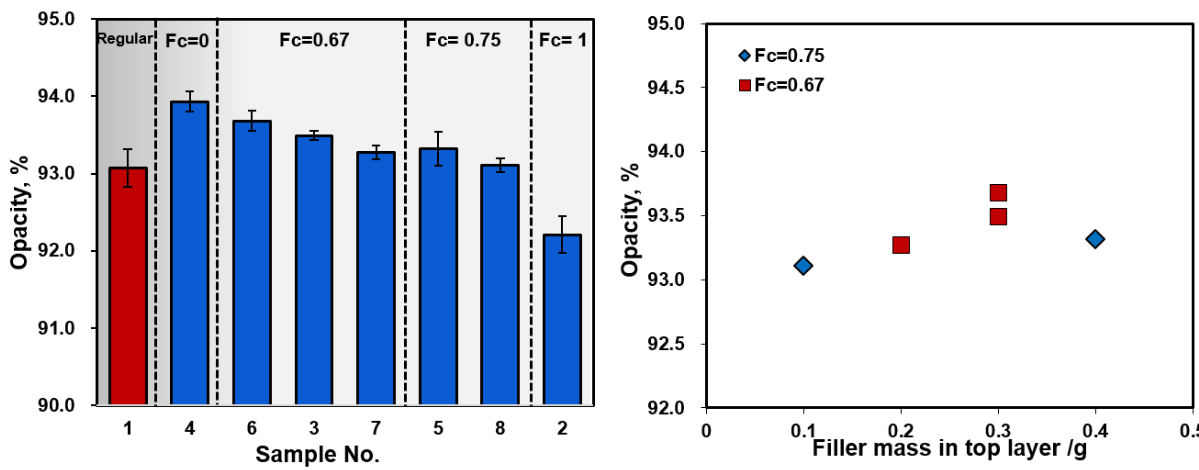

Fig. 5. Effect of filler distribution factor on paper opacity

The results from the strength property test showed that the effect of filler on tensile index was not consistent with the inter-bonding strength for laminated paper. Tensile strength describes the maximum in-plane stress loaded on the paper under a given width of test specimen, and the stress depends greatly on the weakest points in the paper matrix. When filler was concentrated in the middle layer, the weakest points were most likely found in the middle layer compared to other sheet layers. In the process of stress loading, the breaking point would likely be found in the middle layer. As the failure happened in the middle layer, the tensile stress would be transferred to other layers. Thus, more force was needed to be loaded because there was no filler in both surface layers, which resulted in a higher tensile index in sample 2. As F_c was decreased, *i.e.*, filler distribution became even, the weak point in each layer would become similar, and the force for breaking paper was reduced. Differing from tensile index, inter-bonding strength describes bonding strength between fibers out of the plane. Filler particles concentrated in one layer would remarkably weaken the bonding strength between sheet layers. During inter-bonding strength test, it was observed that the fracture area always occurred in the layers with higher filler content.

In the paper industry, paper with a layered structure, such as linerboard, can be laminated from different headboxes. F_c can be used as a useful tool to optimize the paper properties. In the literature, an inter-filling method, which was developed by Han *et al.* (2013), endowed paper with superior tensile strength compared to the traditional filling method. As for this method, filler particles were added between the wet layers, and the strengthen aids, such as cationic starch, were applied in the filler layer to increase the inter-bonding strength of laminated paper.

Optical Properties

The addition of mineral fillers generally increases paper optical properties which are influenced by the filler type, characteristics, and distribution in paper. (Koivunen and Paulapuro 2010; Seo *et al.* 2012*;* Adel *et al.* 2016). As shown in Fig. 4A, the brightness of the laminated paper can be varied from 86 to 87.1% ISO for a given filler content. The increase in filler content of the top layer helped to improve paper brightness because the filler content on the surface of the paper was also increased. In addition, for a given filler content in the top layer, the increase in filler in the middle layer can help improve reflectance efficiency and thus, increase the brightness. This can be verified from sample 3 versus 6 and 7 versus 4.

There was no obvious relationship between F_c and paper brightness, as shown in Fig. 4B. For a given F_c, higher filler content in the surface layer gave a higher brightness. Additionally, the sidedness in brightness can be also observed. The brightness of the top side is typically higher than that of the bottom side due to increased loss of filler particles in the bottom side during drainage (Odell 2000). Based on this, the correlation between the sidedness in brightness and characteristics of filler distribution was analyzed. However, only the symmetry factor, F_{sym}, which proposed by Puurtinen (2004), was highly related to paper sidedness, as shown in Fig. 4C. The sidedness in brightness can be alleviated by optimizing the drainage conditions or controlling the filler distribution on both sides of the sheet.

Besides the improvement of brightness, the use of filler can also produce fiber-air-filler interfaces and increase the light scattering, resulting in enhanced paper opacity. Filler distributed in the cross section of paper can influence fiber-air-filler interfaces, and thus, increase the paper opacity. A good relationship can be found between F_c and paper opacity, as shown in Fig. 5A. Enhanced uniformity of filler distribution in the cross-section of paper, *i.e.*, decreasing F_c, can improve paper opacity. Figure 5B indicates the higher filler content in the top layer, the better opacity can be obtained for a given F_c. When filler particles were concentrated in one layer, the particles were packed together simultaneously, which resulted in the enhanced bonding between fibers and decreased the light scattering area of fillers. The decrease of paper bulk and the increase of tensile index can also partly explain the loss of opacity. Therefore, even though the filler content was constant, the optical properties of the filled paper can still be further optimized by controlling the filler distribution characteristics.

CONCLUSIONS

1. A new filler distribution factor (F_c) was proposed to characterize filler particles concentrated in the z-direction of laminated paper for a given filler content. F_c was demonstrated to be an effective tool to optimize paper properties.
2. The increase in F_c can decrease paper bulk and porosity of the filled paper. When filler particles were concentrated in one layer of laminated paper, *i.e.*, $F_c = 1$, the bulk of the filled paper decreased 11.3% while the air resistance of the paper increased 3.8 times compared to the paper with $F_c = 0$.
3. The tensile index of the filled paper was enhanced when fillers concentrated in the middle layer or filler content was increased in the surface layer for a given F_c. When $F_c = 1$, the tensile strength increased between 26 to 40% compared to that of the other samples. However, the negative effect of filler addition on inter-bonding strength of filled paper was pronounced as F_c was increased.
4. Paper brightness was largely affected by filler concentration on the paper surface. Increasing the uniformity of filler distribution in the z-direction of the paper contributed to paper opacity.

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