# LUMEN LOADING WITH CALCIUM CARBONATE PIGMENTS

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Keywords: Cellulose, Fiber, Papermaking, Pigment, Structure.

Abstract: Lumen loading is a method that retains colloidal pigments into the porous structure of cellulosic fibers. It has been reported that lumen loading can provide better paper properties compared with loading by direct addition of pigments into the papermaking furnish. The paper properties, including optical, physical, surface and structural, can in turn affect the paper printability. The objectives of this research are: to load calcium carbonate pigments into the fiber pores as much as possible, to compare the lumen loading results from a mechanical method and a chemical method, and to evaluate the properties of the handsheets made from lumen loaded fibers. Calcium carbonate pigments are studied because of the current interest in the acid-to-alkaline conversion of the papermaking chemistry. Experimental results indicate that the greatest lumen loading levels are 10.8% by the mechanical method and 13.0% by the chemical method. Lumen loading with the mechanical method are mainly affected by mixing speed, mixing time, and the particle size of calcium carbonate. However, lumen loading with the chemical method are primarily controlled by the dosage level of the precipitating chemicals, sodium carbonate and calcium hydroxide, and the molar ratio of the two chemicals. Compared with the direct addition handsheets at equivalent loading levels, the lumen loaded handsheets have significantly higher brightness, opacity, and tear resistance. However, the lumen loaded handsheets reveal lower smoothness, gloss, and bursting strength.

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## Introduction

Cellulosic fibers are the major component of different papers. Their dimensions are 1-5 mm long and 0.01-0.05 mm wide. Their density ranges from 0.3 to 0.7 g/cm$^3$. These fibers have a porous structure and the center cavity is named lumen. The pore diameters have been reported as from 5 to 300 Å (Ko, 1981). The specific surface area of the pores can be as high as 1,000 m$^2$ (Stone and Scallan, 1968), and the pore volume is about 2 ml/g fibers (Allan, et al., 1991). These pores have been used as a locale for retaining inorganic pigments, and the retaining method is called lumen loading (Haslam and Steele, 1936; Green, *et al.*, 1982; Miller and Paliwal, 1985). Comparing to the retention outside the pores, pigments retained within the pores can provide enhanced paper properties as discussed below.

## Effects of lumen loading

The reasons of making paper with inorganic pigments, such as clay, calcium carbonate, and titanium dioxide, are to improve the physical, optical, price-performance, and esthetic properties of the finished paper (Gill and Hagemeyer, 1992). However, due to their colloidal dimensions being considerably smaller than the opening of the paper forming wire, these pigments usually experience a low retention, thus decreasing the efficiency of material application. Moreover, the colloidal particles can block the bonding sites between the cellulosic fibers, causing decreased tensile strength of the end paper (Alince and Lepoutre, 1985). Furthermore, the use of polymeric flocculants for increased pigment retention would decrease the dispersing state of pigment particles, resulting in lowered optical performance.

Lumen loading, by retaining pigment particles into the fiber pores before making paper, can potentially resolve the technical problems stated above. This is because lumen loaded particles are shielded by the pore structure and would not be sheared away from the pores when making paper, and thus an increased pigment retention. Lumen-loaded particles would not block the fiber-to-fiber bonding as well, since this type of bonding occurs only on the fiber surface, hence an improved bonding magnitude. Finally, since the particles are lumen loaded before the application of the flocculants, the dispersing state should remain in the final sheet of paper giving improved optical properties. Table 1 lists five technical advantages of lumen loaded paper (Chang, 1996).

Table 1. Potential technical advantages of lumen loaded paper.

| |
|---|
| Increased brightness |
| Increased opacity |
| Increased mechanical strength |
| Improved pigment retention |
| Improved material distribution in *z* direction |

## Methods of lumen loading

Two different methods have been used for lumen loading: mechanical method and chemical method. The mechanical method uses high shear and centrifugal forces to load the pigment particles into the fiber pores. The loading mechanism was reported as an adsorption process (Scallan and Middleton, 1985), therefore the time rate of loading is a function of the pigment concentration. Moreover, the surface charges on the fiber and the pigment particles should be opposite for increased attractions between them. To determine the loading results by the mechanical method, a washing step is performed to eliminate particles outside the pores followed by ashing the loaded fibers. And scanning electron microscope (SEM) is used to produce photographic evidences for successful lumen loadings.

Although a limiting factor for the mechanical method is that the particle size of the pigment has to be small enough for the penetration and adsorption to occur, the chemical method is not limited by this factor. Chemical method starts with mixing and soaking the fibers with a soluble salt. Then, the fiber-salt suspension is added into another salt solution. Through chemical reaction, the pigment particles are precipitated *in situ* within the fiber pores. For example, calcium hydroxide was mixed and soaked with the fibers, then the suspension was added into a sodium carbonate solution. The calcium carbonate particles were precipitated and present *in situ* within the fiber pores (Allan, *et al.*, 1992). The determination procedure of the loading results is the same as the mechanical method including washing, ashing, and SEM analysis. The influencing factors for the chemical method are those associated with the precipitation reaction such as salt concentration, pH, and temperature.

Although both mechanical and chemical methods have been reported in the literature, no information could be found about the comparison between the two methods using the same cellulosic fibers in one research project. This research loads calcium carbonate pigments into the fiber

pores and compares the results generated from the mechanical method with the chemical method. Calcium carbonate pigments were used because of the current interest in the acid-to-alkaline conversion of the papermaking chemistry (Scott, 1992).

## Research objectives

The objectives of this research are: to load calcium carbonate into the fiber pores as much as possible, to compare the lumen loading results from the mechanical method with that from the chemical method, and to evaluate the properties of the handsheets made from lumen loaded fibers.

# Experimental procedures

## General procedures

The cellulosic fibers used in this research were fully bleached, never-dried pulp composing E-leijhou (42.0%), Birch (21.4%), Beech (25.6%), and Mangrove (11.0%). The pulp was beaten in a Valley beater (1.57%) to various degrees of CSF (Canadian Standard Freeness). The beaten pulp was then washed to remove the fines through a filtration (100 mesh) step. The washed pulp was next used for lumen loading under different mechanical and chemical conditions. After the lumen loading step, the pigment particles outside the fiber pores were eliminated through another washing step. This washing step was performed until the clearness of the filtrate was the same as the washing liquor (distilled water). Material balances were practiced for the calcium carbonate with the pulp and in the washing filtrate. Distilled water was used for all experimental steps. The lumen loaded pulp was finally dried (105 °C) and ashed (925 °C) to determine the quantity of lumen loading.

British Standard Handsheet Mold was used to make handsheets (60 $g/m^2$). The pulp was diluted (0.5%) and dispersed using a magnetic stirrer before adding to the sheet mold. For making handsheets with directly added calcium carbonate, a low-charge-density, high-molecular-weight, polyacrylamide (0.15% addition level) was applied. The handsheets were pressed and conditioned (20 °C, 65% humidity) before the property testing. Tappi standard methods were followed to obtain data on the optical, surface, and mechanical properties of the handsheets. An SEM (Hitachi, S-2400) was used to obtain photographs of the handsheets.

Lumen loading procedures

For the mechanical method, the pulp (10 g) was first slurried (920 ml water) using a three-blade mixer. Then, a low-molecular-weight, high-charge-density polyethylenimine (PEI, 1% solution) was added to the pulp slurry and gently mixed for 5 min. Next, calcium carbonate suspension (10 g, 6.7% ) was metered into the pulp slurry. The starting calcium carbonate was a precipitated variety with an average particle size of 0.48 μm. The mixed slurry was diluted (1.6%) with distilled water. The pH of the slurry was then adjusted. The mixing speed (rpm) was afterward adjusted for a pre-determined time period. After these steps, the pulp was washed dried, and ashed as stated above.

Six variables were investigated for their effects on the outcomes of lumen loading by the mechanical method . These variables are polymer (PEI) treatment, pulp freeness (CSF), pH at the lumen loading step, mixing speed (rpm), mixing time (min), and average particle size of calcium carbonate. These variables were studied in the above sequence, and the next variable was studied using the best outcome obtained from the previous variable. The PEI treatment was performed at the dosage levels from 0 to 10%; CSF levels were from 200 to 600 ml; the pH at the lumen loading step was adjusted between 8 and 13; the mixing speed was between 500 and 4,500 rpm and the mixing time was from 20 to 120 min; and the average particle size of calcium carbonate pigments ranged from 0.12 to 1.17 μm.

For the chemical method, the pulp (10 g, CSF 506 ml) was first slurried with water. Then, the sodium carbonate solution was measured into the pulp slurry with a gentle mixing for 5 min. Next, the calcium hydroxide solution was metered into the pulp slurry. Afterward, the pulp slurry was adjusted for its pH (13.0) and then stirred (1,500 rpm) for a pre-determined time period. Finally, the pulp slurry with precipitated calcium carbonate was washed, dried, and ashed following the steps stated above.

Four variables were studied for the chemical method of lumen loading. Theses variables are chemical dosage, chemical's molar ratio, mixing time, and chemical's order of addition. As in the mechanical method, these variables were studied in the stated sequence, and the next variable was studied using the best outcome obtained from the previous variable. The chemical dosage was adjusted between 0.02 and 0.15 molar as the final concentration; the chemical molar ratio was from 1/5 (sodium carbonate/calcium hydroxide) to 5/1; the mixing time was either 20 or 40 min; and the order of addition was sodium carbonate first calcium hydroxide

second, calcium hydroxide first sodium carbonate second, or adding the two chemicals together into the pulp slurry.

## Results and discussion

### Mechanical method

Six variables were studied to evaluate their effects on the outcomes of lumen loading. These variables are polymer treatments, pulp freeness, pH, mixing speed, mixing time, and average particle size. They were experimented following the above stated sequence. Figure 1 shows the effect of the polymer treatment, the first variable. The polymer used was a low-molecular-weight, high-charge-density cationic polyethylenimine (PEI). When running the PEI experiment, the other five variables were kept constant with pulp freeness being 602 ml (CSF), pH at the lumen loading step being 8.0, mixing speed being 3,000 rpm, mixing time being 20 min, and the average particle size of calcium carbonate being 0.48 μm.

Figure 1 shows that without PEI addition, some calcium carbonate can be loaded (1.24%) into the pores of the cellulosic fibers. As the addition

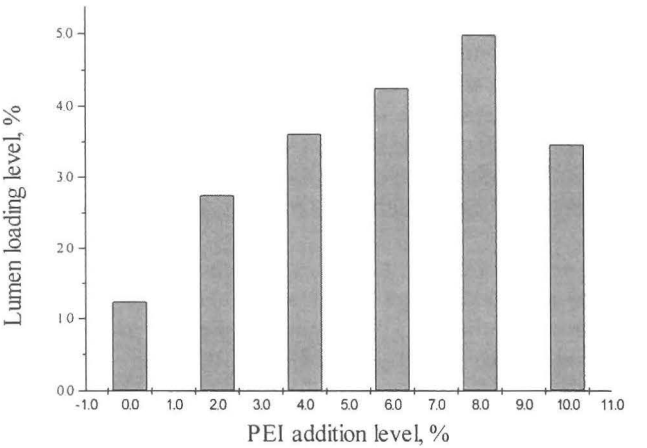


Figure 1. The effect of PEI on lumen loading.

level of PEI increases, the magnitude of lumen loading keeps increasing up to 4.98%, then decreases down to 3.46%. Apparently, without PEI the lumen loading is effected by the shear and centrifugal forces that transport the pigment particles into the fiber pores. With the PEI treatment, however, the increased lumen loading should be a result of the charge modification on the fiber surface. The cellulosic fibers containing carboxylate ($-COO^-$) groups give them a negatively charged surface after ionization. This charge could be neutralized or be even reversed after the adsorption of the cationic PEI. The charge reversal can in turn increase the attraction between calcium carbonate and the fiber surface (also the pore surface), resulting in higher lumen loading. The plausible interpretation for the decrease in lumen loading at 10 % PEI addition is an over dosage of PEI. Over dosage might cause the surface charge of calcium carbonate to become positive, rendering a repulsion between the pigment particles and fiber surface. Therefore, PEI treatment on fibers has a direct effect on the lumen loading, but only to an extent. And an over dosage of PEI can lead to a decreased lumen loading due to charge repulsion.

Figure 2 suggests that pulp freeness (CSF) can affect the outcomes of lumen loading as well. It seems that a linear relationship is present between the CSF and lumen loading levels, even though the difference in lumen loading is merely about one percent. The fact that the lower the CSF, the lesser the lumen loading implies that mechanical treatment on fibers probably has collapsed the fiber pores to some degree. This pore collapsing might decelerate the transporting process of pigment particles moving into the pores, thereby decreasing the final levels of lumen loading.

For the four other variables including pH at the lumen loading step, mixing speed, mixing time, and average particle size of calcium carbonate, significant differences in the magnitude of lumen loading were also found. However, the experimental data did not reveal a systematic pattern (Chang, 1996), and their effects on the outcomes of lumen loading are discussed here using Table 2. Table 2 lists the maximum differences in the final levels of

Table 2. The maximum differences in the final levels of lumen loading affected by the mechanical variables.

| Mechanical variable | Maximum difference, % |
|---|---|
| mixing speed | 6.99 |
| mixing time | 6.72 |
| average particle size | 6.19 |
| PEI treatment | 3.74 |
| pH | 2.24 |
| pulp freeness | 1.15 |

lumen loading affected by all six mechanical variables studied in this research, and the differences are ordered from the greatest to the least.

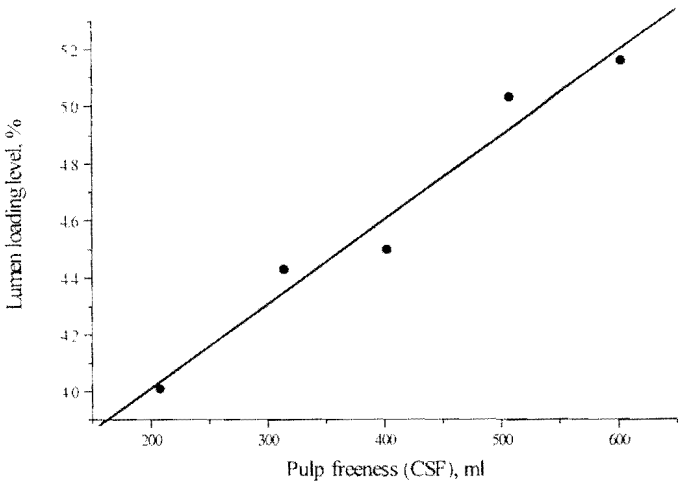


Figure 2. The effect of pulp freeness on lumen loading.

When the pH at the lumen loading step was studied, the pulp had a freeness (CSF) of 606 ml and PEI treatment at 8%. The lumen loading results were increased from 5% at pH 8.0 to 7.24% at pH 13.0. This is possibly because a higher pH increases both the ionization on the fiber surface and PEI, providing a better charge modification needed for the inward transport of the pigment particles. In addition, the fibers are probably more swelled as the pH increases, facilitating the particles to move toward and into the pores.

The results of mixing speed denote that there is an optimum mixing speed at 3,000 rpm for a maximum lumen loading of 10.8%. In other words, either lower mixing speeds (500 and 1,000 rpm) or higher mixing speed (4,500 rpm) can not enhance the lumen loading. For the mixing time, similar results were obtained, *i.e.*, extending the mixing time up to 120 min can not bring about higher lumen loading levels. These findings suggest that slower mixing speed would decrease the shear and centrifugal forces for accomplishing the transport of the pigment particles. And too strong mixing speed might have resulted in a reversed movement of the particles,

causing an outward transport from the interior of the pores. Similarly, too long mixing time would also cause an outward transport of the pigment particles, and thus lowered lumen loading.

The final variable studied was the average particle size of calcium carbonate. It was found that an average particle size of 0.48 μm gave the greatest lumen loading of 10.8%. Smaller average size of 0.12 μm yielded a loading level of 5.64% and larger particle size of 1.17 μm afforded a level of 6.34%. Thus, it is postulated that smaller particle size can move outward away from the pores and larger particle size can experience a harder environment advancing into the pores, thereby decreasing the lumen loading.

In summary, all six variables studied have an effect on the outcomes of the lumen loading. And their effectiveness of varying the loading outcomes follows an order of mixing speed, mixing time, average particle size, polymer treatment, pH at the lumen loading step, and pulp freeness (Table 2). The greatest lumen loading accomplished is 10.8% applying the conditions of mixing speed at 3,000 rpm, mixing time at 20 min, average particle size at 0.48 μm, PEI treatment at 8%, pH at the lumen loading step being 13.0, and finally pulp freeness (CSF) at 606 ml.

## Chemical method

The chemical method of lumen loading studied was based on the precipitation reaction (1) occurring between sodium carbonate ($Na_2CO_3$) and

$$Na_2CO_3 + Ca(OH)_2 \rightarrow CaCO_3 + 2\ NaOH \qquad (1)$$

calcium hydroxide [$Ca(OH)_2$]. The ionic species of carbonate ($CO_3$ ) was first well mixed with the fibers. Then another ion ($Ca^{+2}$) was introduced into the fiber slurry for the precipitation of calcium carbonate. Via this procedure, calcium carbonate pigment was produced *in situ* inside the fiber pores. Four variables were studied to evaluate their effects on the lumen loading outcomes: chemical dosage, chemical's molar ratio, mixing time, and the order of addition of the precipitating chemicals.

Figure 3 delineates the effect of chemical dosage on lumen loading. Apparently, adding 0.02 moles of sodium carbonate and 0.02 moles of calcium hydroxide into the pulp slurry can stoichiometrically produce 2.0 g calcium carbonate. But increasing their dosage to 0.15 moles should yield 15 g calcium carbonate and an increased probability of precipitating calcium carbonate inside the fiber pores, resulting in greater lumen loading.

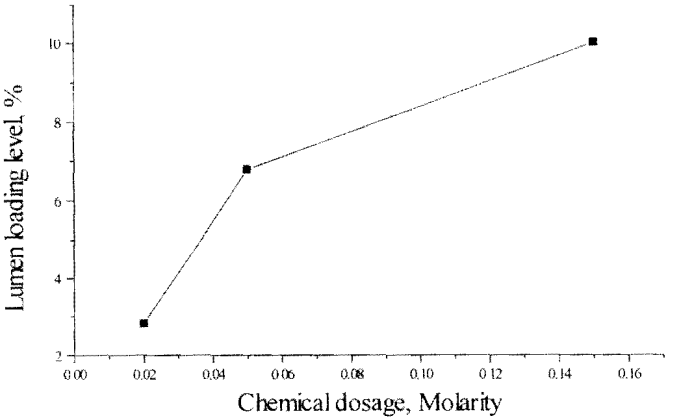


Figure 3. The effect of chemical dosage on lumen loading.

After varying the molar ratio of the chemicals added into the pulp slurry, Figure 4 suggests that the greatest lumen loading occurs at a molar ratio of one. Either increasing the molar amount of sodium carbonate or calcium hydroxide decreases the final outcomes of lumen loading. Moreover, the greater the difference in molar ratio, the lower the outcome. Furthermore, higher amount of sodium carbonate than calcium hydroxide can provide relatively lowered lumen loading. These findings imply that excess amount of either chemical would not facilitate the lumen loading. This is probably because the presence of excess ionic species hinders the precipitation process and because an adsorption of the excess ion onto the surface of the already-formed calcium carbonate particles occurs. The hindrance would give lower results as shown in Figure 4. And the adsorption of carbonate ion would provide a negatively charged surface of calcium carbonate. This negative charge can in turn repel the calcium carbonate particles against the pore surface, thereby lowering lumen loading as Figure 4 depicts.

The mixing time studied was either 20 or 40 min, and the lumen loading outcomes were 12.95% and 7.39% respectively. These data imply that longer mixing time might have withdrawn the particles out of the pores into the bulk of the solution, giving decreased result. The last variable studied was the order of addition, *i.e.*, either sodium carbonate or calcium hydroxide was added first into the pulp slurry, or they both were added at

the same time.

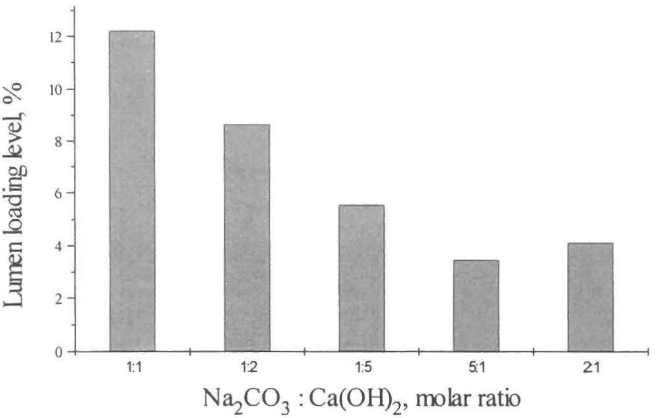


Figure 4. The effect of chemical's molar ratio on lumen loading.

The results are that simultaneous addition yields the least lumen loading (9.92%), probably because more precipitation has occurred outside the pores. And the fact that adding sodium carbonate first gives higher result (13.0%) than adding calcium hydroxide first (9.59%) might have a relation with the particle size of calcium carbonate. In other words, adding sodium carbonate first might produce a particle size that is more suitable for the retention inside the pores than adding calcium hydroxide first. Further investigation on this hypothesis has undertaken in our laboratory.

Thus for the chemical method, the experimental results have shown that the greatest amount of lumen loading (13.0%) occurs when adding sodium carbonate first into the pulp slurry. The molar ratio of the chemicals is one with their concentration being 0.15 molar and the mixing time is 20 min. Table 3 lists the maximum differences in the final levels of lumen loading affected by the chemical variables.

Properties of lumen loaded handsheets

Lumen-loaded handsheets were made following the procedures of

the chemical method with controlled loading levels. Their properties are

Table 3. The maximum differences in the final levels of lumen loading affected by the chemical variables.

| Chemical variable | Maximum difference, % |
|---|---|
| molar ratio | 8.75 |
| chemical dosage | 7.08 |
| mixing time | 5.56 |
| order of addition | 3.36 |

compared with the handsheets made by direct addition of calcium carbonate (average particle size of 1.0 μm) into the pulp furnish. A low-charge-density, high-molecular-weight polyacrylamide was used as the retention aid (flocculant) for controlling the pigment loading level.

Figures 5 to 7 are the SEM photographs depicting handsheets without calcium carbonate, with direct addition of calcium carbonate, and with lumen loaded calcium carbonate. Evidently, without the presence of calcium carbonate, the fiber surface is clean and the pores are empty (Figure 5). And Figure 6 reveals that when calcium carbonate was loaded (7.76%) by the direct addition method, pigment particles are deposited on the fiber surface not inside the pores. However, when calcium carbonate was loaded (7.89%) by the chemical method, the pigment particles are all retained inside the pores, leaving the fiber surface clean. Thus, contrasted to the direct addition of calcium carbonate, lumen loading selectively retains the pigment particles into the fiber pores and leaves a clean surface of the pulp fibers.

Lumen loading can improve the brightness and opacity of the handsheets, as Figures 8 and 9 illustrate. Comparing at similar loading levels, the lumen loaded handsheets have higher brightness and opacity. This effect is seemingly augmented as the loading level increases. These experimental results are apparently due to the dispersion state of the pigment particles. When making handsheets by the direct addition method, a polymeric retention aid was used. This would cause a significant flocculation among the pigment particles and the pulp fibers, reducing the amount of air-pigment, air-fiber, and fiber-pigment interfaces. These interfaces are needed for the light scattering to improve the handsheet's brightness and opacity. On the other hand, when making handsheets by the lumen loaded fibers, no retention aid was used, and the pigment particles would remain well dispersed, giving rise to higher brightness and opacity. Nonetheless, lumen loading did result in lower handsheet gloss as shown in

Figure 10. This is probably because the direct addition of calcium carbonate can fill the holes between the fibers, affording handsheets with a smoother surface as illustrated by Figure 11. Thus, the selective retention of the pigment particles inside the fiber pores would effect a relatively coarse surface structure, and then a lowered gloss.

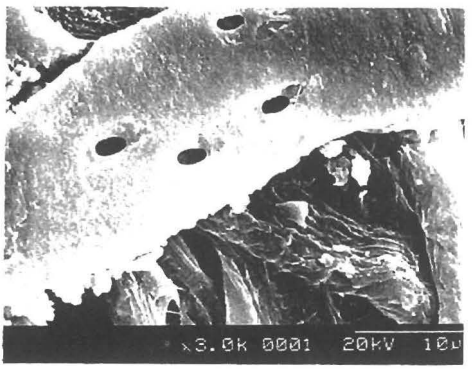


Figure 5. SEM photograph (3,000x) of a handsheet without calcium carbonate pigment.

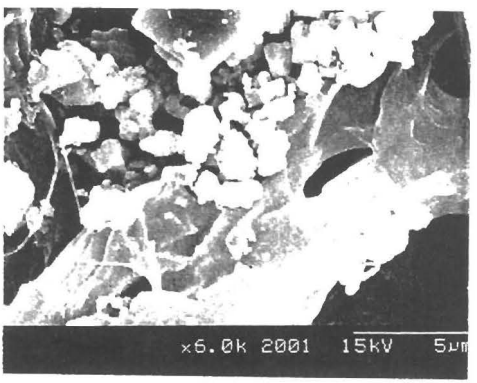


Figure 6. SEM photograph (6,000x) of a handsheet made from direct addition of calcium carbonate pigment.

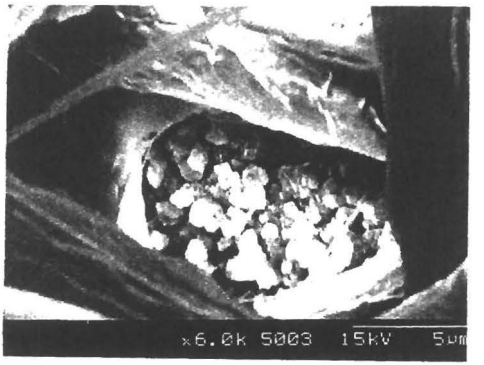


Figure 7. SEM photograph (6,000x) of a handsheet made from the chemical loading of calcium carbonate pigment.

With respect to the strength properties, lumen loaded handsheets should provide greater results than that from the direct addition handsheets. The plausible reason is that lumen loaded handsheets would not have a blocking effect on the interfiber bonding which is a significant factor for developing sheet strength. This debonding interpretation is supported by Figure 12 showing that the tear index of the lumen loaded handsheets is

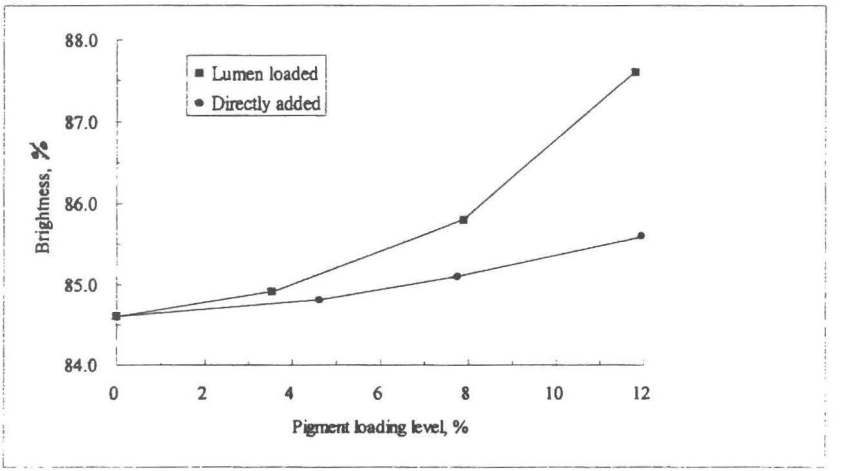


Figure 8. The effect of lumen loading on handsheet brightness.

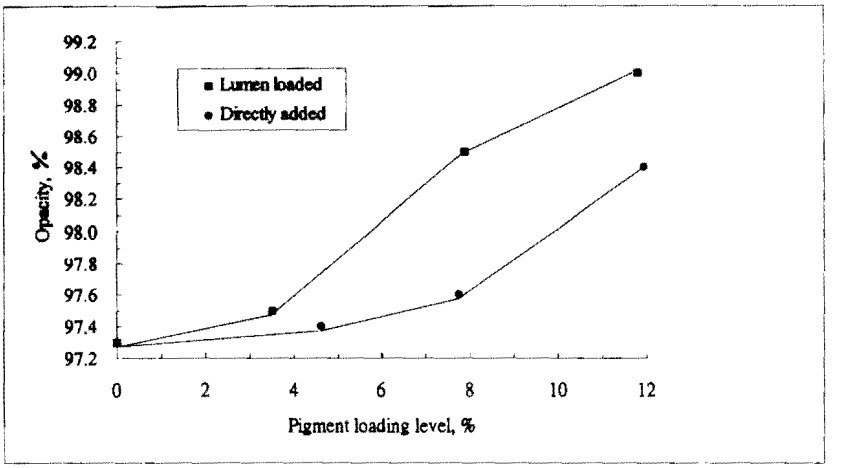


Figure 9. The effect of lumen loading on handsheet opacity.

greater at all tested loading levels than the direct addition handsheets. In addition, as the loading level increases, the debonding effect becomes more evident, since the difference in tear resistance increases with the loading level. However, Figure 13 illustrates that the tensile strength did not display a significant difference until the loading level reached 12%. This suggests that the tensile strength is not only dependent on the interfiber

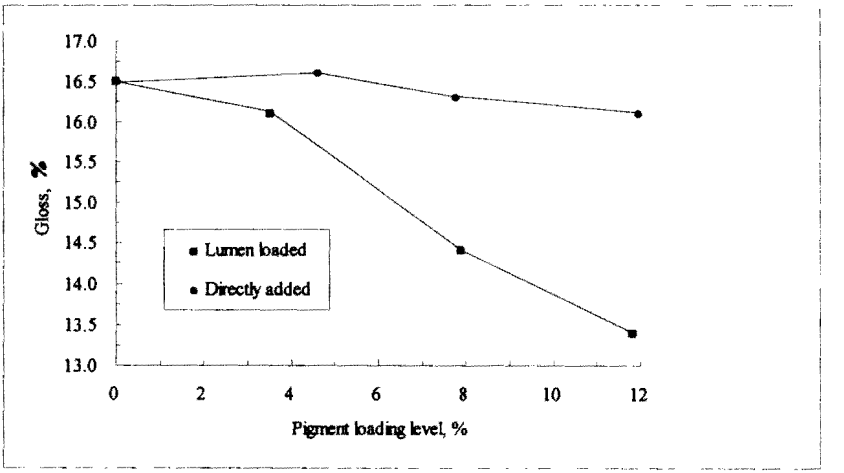


Figure 10. The effect of lumen loading on handsheet gloss.

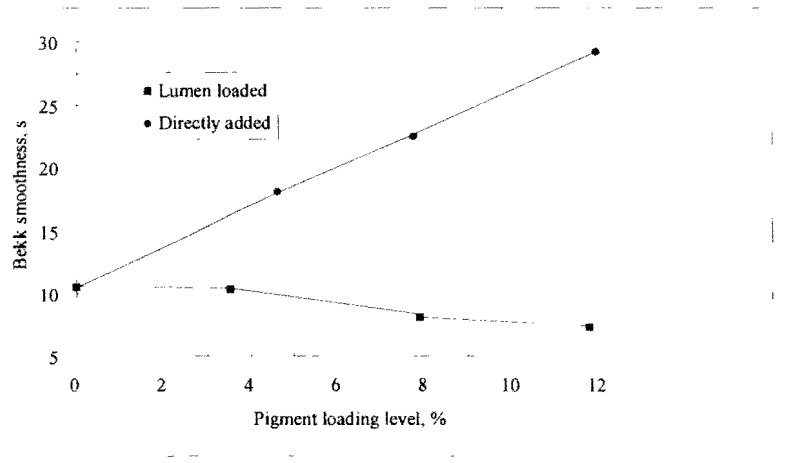


Figure 11. The effect of lumen loading on handsheet smoothness.

bonding, but on the strength nature of the fibers themselves, such as the fiber flexibility (Steadman and Luner, 1985). It is plausible that at the higher lumen loading level, the fiber flexibility might have decreased to a meaningful extent, providing lesser twisting and weaving of the fibers in handsheets which then lowers the tensile strength. A decrease in fiber flexibility is in agreement with the results of bursting strength which is

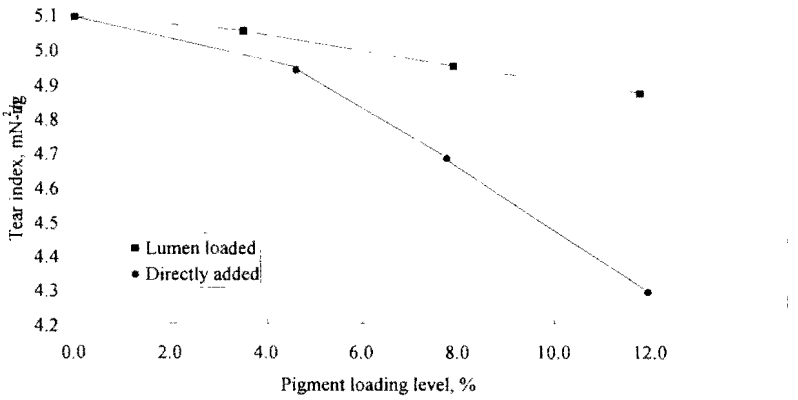


Figure 12. The effect of lumen loading on the tear resistance of handsheets.

greatly dependent on the fiber flexibility. As clearly shown in Figure 14, the burst index with the lumen loaded handsheets is lower than that with the direct addition handsheets at all tested loading levels.

Hence, it can be concluded that the strength properties of the handsheets are significantly modified by the lumen loading. Lumen loading, without the blocking effect on the interfiber bonding, results in greater tear resistance. However, since lumen loading might decrease the fiber flexibility, it can decrease the bursting strength and the tensile strength at a higher loading level.

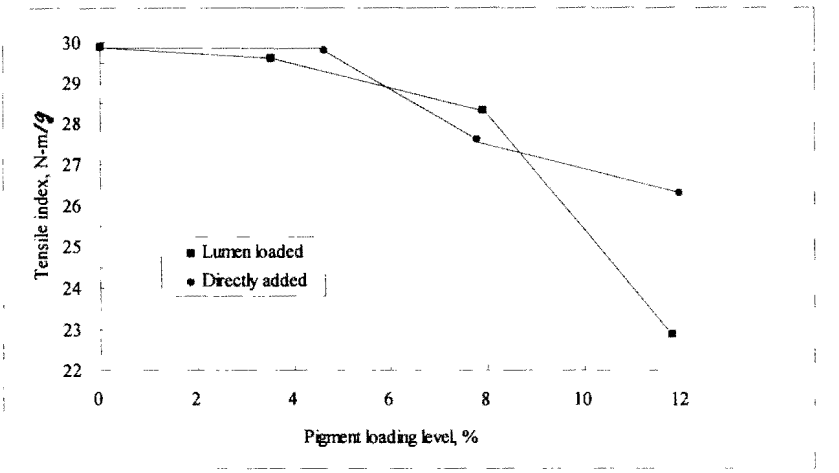


Figure 13. The effect of lumen loading on the tensile strength of handsheets.

Additionally, a simple calculation assuming the void volume of the fibers is 2 ml/g fibers, the handsheet weighs 1.2 g containing 12% pigment, and the precipitated calcium carbonate has a density of 2.7 g/cm$^3$, less than 3% of fiber void volume is occupied by the calcium carbonate particles. This implies that more calcium carbonate could be loaded into the fiber pores. Finally, what printability of the lumen loaded fibers can provide is a significant area to research.

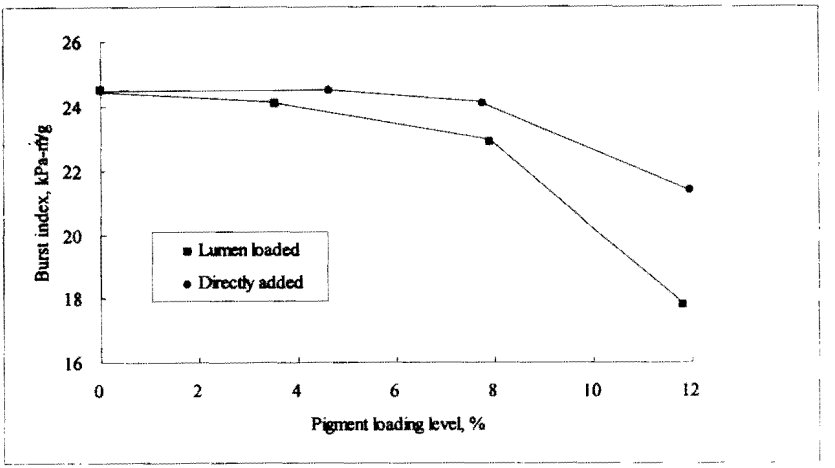


Figure 14. The effect of lumen loading on the bursting strength of handsheets.

## Conclusions

Both mechanical and chemical methods can provide lumen loaded fibers. The greatest loading level with the mechanical method is 10.8% with the mixing speed as its main controlling variable. For the chemical method, the greatest loading level is 13.0% controlled chiefly by the molar dosage of the precipitating chemicals. Lumen loaded handsheets have higher brightness and opacity, probably because the particles are better dispersed. However, lumen loading provides a coarser surface structure, and thus lower smoothness and gloss. Finally, lumen loading significantly affects the strength properties of the handsheets. Without blocking the interfiber bonding, lumen loaded handsheets consistently have greater tear resistance. Nonetheless, lumen loading can effect lowered bursting strength or tensile strength at higher levels, possibly due to a decrease in the fiber flexibility after the lumen loading procedures.

## Acknowledgments

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