A PRELIMINARY INVESTIGATION INTO THE USE OF ACID-TOLERANT PRECIPITATED CALCIUM CARBONATE FILLERS IN PAPERMAKING OF DEINKED PULP DERIVED FROM RECYCLED NEWSPAPER

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The use of acid-tolerant precipitated calcium carbonate fillers, including phosphoric acid/sodium hexametaphosphate modified precipitated $CaCO_3$ filler, and sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated $CaCO_3$ filler in papermaking of deinked pulp derived from recycled newspaper was explored. These two acid-tolerant fillers provided considerably more brightness improvement in papers in comparison the unmodified filler, presumably indicating alleviated pulp darkening achieved as a result of better acid-resistant properties. The addition of acid-tolerant fillers into the furnish slurries gave lower system pH as compared with unmodified filler. Among the three fillers used in this work, the effect on retention of modification of the filler with sodium silicate/phosphoric acid/sodium hexametaphosphate was probably the best, as evaluated from ash content measurements. For air permeability of the paper, the use of acid-tolerant fillers provided slightly more improvement in comparison to the unmodified filler. For tensile and burst strength of the paper, the use of sodium silicate/phosphoric acid/sodium hexameta-phosphate modified precipitated calcium carbonate filler gave better results as compared with the other two fillers. Additionally, the improving effect of acid-tolerant fillers on furnish static drainage was found to be slightly weaker than that of unmodified filler.

Keywords: Acid-tolerant precipitated calcium carbonate filler; Papermaking; Deinked pulp; Optical properties; pH profile; Strength properties; Air permeability; Ash content; Furnish static drainage

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INTRODUCTION

Calcium carbonate fillers have been widely used in woodfree paper grades since the 1980's to improve the optical properties of paper products and to reduce manufacturing costs. In woodfree paper grades, the alkaline nature of calcium carbonate is an additional benefit because it creates a stable buffered system and provides some paper strength improvement (Evans and Slozer 2003), and paper products filled with calcium carbonate usually have better aging resistance as compared with other fillers (Gill and Scott 1987). In the manufacture of paper there is also an economic incentive to use calcium carbonate fillers because of the ability to produce cheap precipitated calcium carbonate on-site at the paper mill (Pang 2001). However, the use of calcium carbonate fillers in wood-containing paper is generally considered as not industrially feasible because of pulp darkening at alkaline pH and dissolution below pH 7 (Ain and Laleg 1997; Evans and Drummond 1991), and their use in rosin-sized paper has also many limitations due to saponification reaction of rosin sizes under alkaline conditions (Shen et al. 2007). The alkaline nature of calcium carbonate fillers and their poor acid-resistant properties make them unsuitable for use in acid and pseudo-neutral papermaking. Titanium dioxide and calcined clay have often been used as fillers in acid and pseudo-neutral papermaking. However, these materials, and titanium dioxide in particular, are expensive, thus adding to the cost of the paper. Talc fillers can also be used in acid and pseudo-neutral papermaking, and the price of talc is generally equivalent to calcium carbonate, but calcium carbonate is much superior to talc in many respects, such as the optical properties of the filled paper. Therefore, there is a need to enable the use of calcium carbonate fillers in acid and pseudo-neutral papermaking.

The methods now available for making calcium carbonate fillers suitable for use in acid and pseudo-neutral papermaking can be divided into the following two categories:

- Addition of a dissolution inhibitor such as carbon dioxide or phosphoric acid (or a combination of various inhibitors) to the filler-containing papermaking wet-end system to inhibit filler dissolution
- Modification of calcium carbonate fillers before use to inhibit filler dissolution and to improve their acid-resistant properties

These two categories of methods have been used in the past few years to obtain acid-tolerant calcium carbonate fillers. From a worldwide perspective, the use of acid-tolerant calcium carbonate fillers in wood-containing paper grades has already been commercialized to a certain degree. In specific industrial applications the use of acid-tolerant calcium carbonate fillers can involve the separate addition of calcium carbonate and dissolution inhibitor or weak acid to furnish. This allows much more effective and efficient pH control of the wet end. It should be mentioned that the use of carbon dioxide to adjust the pH and to inhibit the dissolution of calcium carbonate fillers in papermaking has been a hot topic (Pakarinen and Leino 2001). It has been claimed that the effect of the carbon dioxide is due to the increased amount of carbonate ions which result from the dissolution of carbon dioxide in the aqueous medium, and these carbonate ions affect the balance of the dissociation equation of calcium carbonate in such a way that calcium carbonate has a lower tendency for dissolving and dissociating (Laurila-Lumme et al. 2003).

For filler modification to improve acid-tolerant properties and lower dissolution tendency, there have been considerable reports in the literature, and the modifiers that have been found to be effective can include aluminum salt (Wu 1997a), calcium-chelating agent/weak acid (Passaretti 1991), weak base/weak acid (Wu 1997b) , calcium-salt-based modifiers (Drummond 2001), phosphoric acid (Pang et al. 1998, 2001, 2003; Pang and Englezos 2003), sodium silicate/weak acid (Snowden et al. 2000), zinc compound/silica-containing substance (Tokarz et al. 1991), sodium silicate/zinc chloride/carbon dioxide (Chapnerkar et al. 1992), and anionic starch derivative/cationic starch derivative (Lambert and Lowes 1975). The available filler modification methods can generally be classified into two categories. One is only suitable for use in the production of filler slurries, whereas the other one is capable of producing modified fillers in power form. For example, The calcium-chelating agent/weak acid, and weak base/weak acid systems have been demonstrated as effective in the production of acid-tolerant filler slurries, and the buffering actions of the modifiers can play a decisive role in suppressing the dissolution of calcium carbonate fillers. However, for the use of modifiers such as zinc compound/silica-containing substance in filler modification, the formation of acid-resistant coatings on the filler surfaces contributes significantly to the suppressed dissolution of the modified fillers in acidic mediums, and the modified fillers can exist in powder form.

Phosphoric acid can be used as a modifier of calcium carbonate fillers to inhibit filler dissolution, and its combined use with other modifiers has been shown to be highly effective. Although the use of phosphoric acid in modification of calcium carbonate fillers has certain disadvantages, such as formation of calcium phosphate (scaling), and release of unwanted phosphorus to waste water, it can possibly be used under certain controlled papermaking conditions to enhance the properties of paper and paperboard products. Our previous work has shown that the use of phosphoric acid/sodium hexametaphosphate, or sodium silicate/phosphoric acid/sodium hexametaphosphate in modification of papermaking grade precipitated calcium carbonate filler can inhibit the dissolution of filler in various water solutions with different pH values, and its acid-resistant property can be significantly enhanced (Shen et al. 2008a,b).

In this work, the use of acid-tolerant precipitated calcium carbonate fillers including phosphoric acid/sodium hexametaphosphate modified filler, and sodium silicate/phosphoric acid/sodium hexametaphosphate modified filler in papermaking of deinked pulp derived from recycled newspaper was explored.

EXPERIMENTAL

Materials

Papermaking grade precipitated calcium carbonate filler was obtained from Guangxi Guilin Wuhuan Co., Ltd., China. Deinked pulp derived from recycled newspaper was supplied by Jilin Chenming Paper Co., Ltd., China, and was slightly refined to a beating degree of 57.0 ^{o}SR using a ZQS2 valley beater made in China**.** Cationic polyacrylamide with the trademark of Percol® 47 used as retention aid was obtained from Ciba Specialty Chemicals Co., Ltd., Germany. Phosphoric acid, sodium hexametaphosphate, sodium silicate, and alum were all analytical reagents produced in China.

Acid-tolerant precipitated calcium carbonate fillers were prepared by filler modification. Precipitated calcium carbonate filler slurries with concentration of 10 wt% were initially prepared, and the water solutions of modifiers were then added. The resulting mixtures with PCC concentration of 5 wt% were sufficiently mixed, and were then aged for 24 h before use. The modifiers used for the preparation of the two acid-tolerant fillers were phosphoric acid/sodium hexametaphosphate, and sodium silicate/phosphoric acid/sodium hexameta-phosphate, respectively. The dosages of sodium silicate, phosphoric acid, and sodium hexameta-phosphate were 1 wt%, 4 wt% and 1 wt%, respectively, based on the dry mass of PCC.

EDAX Analysis and SEM Observations of Fillers

EDAX analysis and SEM observations of acid-tolerant precipitated calcium carbonate fillers and unmodified precipitated calcium carbonate filler were performed with a scanning electron microscope with elemental distribution analysis (QUANTA 200).

Evaluation of Influence of Filler Addition on the pH of Furnish Slurry

Filler was added to the pulp slurry to form a suspension with concentration of 0.5 wt/%, and the target filler loading level was controlled to be 15 wt%. The slurry pH as a function of time was measured using a PHS-3C pH meter made in China.

Evaluation of Static Furnish Drainage

The pulp with dry weight of 2 g was mixed with 0.5 g filler to form a 1000 ml suspension, and the freeness (^{o}SR) was measured to evaluate the static drainage property of the filler-containing furnish. The apparatus used was a ZDJ-100 beating degree tester made in China.

Handsheets Preparation

The mixture of pulp and filler was diluted to 0.5 wt%, and the target filler loading level was controlled to be 15 wt%. Cationic polyacrylamide solution was added, and the slurry was stirred for 1 min. Diluted alum solution was then added dropwise to adjust the slurry pH to 6.50, and the pH-adjusting time was controlled to be 1 min. Handsheets with target basis weight of 60 g/m^2 were then prepared using the sheet former (ZQJ1-B200mm) produced in China. The handsheets were further pressed and flattened for 24 h using the apparatus called *Pingyatuo* equipped with the sheet former. The conditions for handsheets preparation were kept consistent for all of the samples.

Measurement of Strength Properties, Optical Properties and Air Permeability of Handsheets

The tensile index values of handsheets were measured using a L&W CE062 tensile strength tester made in Sweden. The burst index values of handsheets were measured using ZDNP-1 burst strength tester made in China. The brightness, opacity and light scattering coefficient of handsheets were measured using a YQ-Z-48A brightness & color tester made in China. The air permeability values of handsheets were measured using YG461E permeability tester made in China.

Measurement of Ash Content of Handsheets

The handsheets were dried at 105 ^{o}C for 6 h, and the dry weight values of handsheets were measured. The dried handsheets were then incinerated at 575 ^{o}C for 6 h, the ash weight values were measured, and the ash content values of handsheets (%) were calculated.

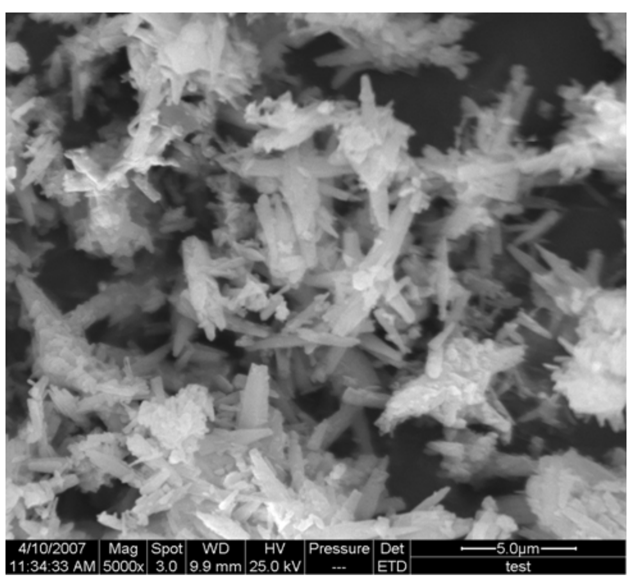
RESULTS AND DISCUSSION

Element Compositions and Surface Morphologies of Acid-Tolerant Fillers

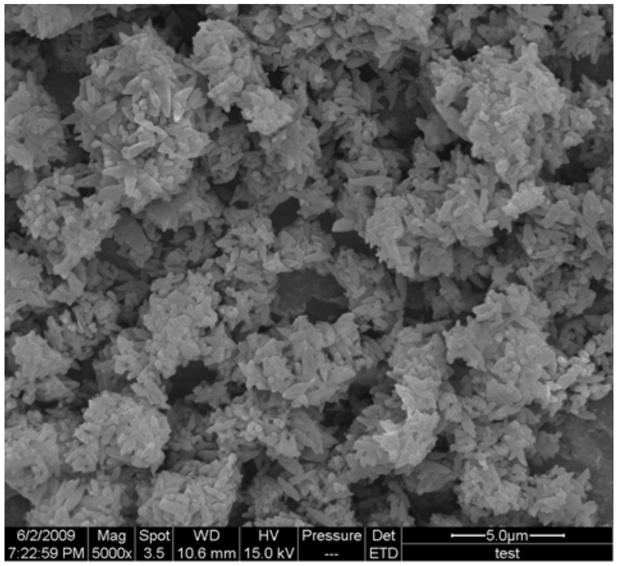
Elemental compositions and SEM images of acid-tolerant fillers and unmodified precipitated calcium carbonate fillers are shown in Table 1 and Fig. 1. It can be seen from Table 1 that the use of phosphoric acid/sodium hexametaphosphate in filler modification might have initiated the precipitation of Ca-P overgrowths on the filler surfaces, and the use of sodium silicate/phosphoric acid/sodium hexametaphosphate in filler modification might have initiated the precipitation of both Ca-P overgrowths and silica coatings. The presence of Ca-P overgrowths and silica coatings on the filler surfaces might act as barriers and block the dissolution sites, which might contribute to the improved acid-resistant properties. It can be seen from Fig. 1 that the morphologies and elemental compositions of acid-tolerant precipitated calcium carbonate fillers were different from that of unmodified precipitated calcium carbonate filler, indicating a change in surface characteristics of filler when modified using the respective modifiers.

Table 1. Elemental Compositions of Acid-Tolerant Precipitated Calcium Carbonate Fillers and Unmodified Precipitated Calcium Carbonate Filler Measured by EDAX.

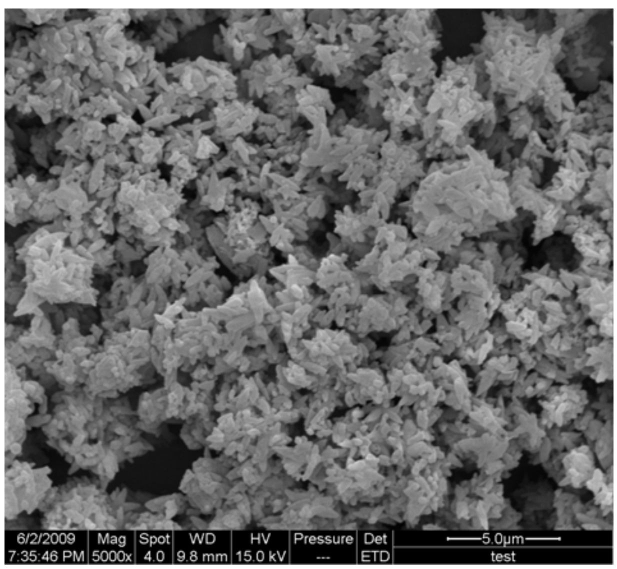
Element	Elemental Content (Molar percent, Atomic)		
	Unmodified Precipitated calcium carbonate filler	Phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler
Carbon	28.57	25.55	25.08
Oxygen	54.24	52.75	52.40
Calcium	16.65	19.57	19.15
Magnesium	0.54	0.65	0.79
Phosphorus	-	1.48	1.95
Silicon	-	-	0.63



(a).



(b)



(c)

Fig. 1. SEM images of different fillers: (a) unmodified precipitated calcium carbonate filler, (b) phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler, and (c) sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler

Influence of Filler Addition on the pH Profile of Furnish Slurry

The influence of addition of different fillers on the pH profile of furnish slurry is shown in Fig. 2. When unmodified precipitated calcium carbonate filler was incorporated into the slurry, the system pH rose to above 9 within 30 minutes. However, the pH values of furnish slurries containing the acid-tolerant precipitated calcium carbonate fillers were quite low (around 7.5). The pH-reducing effect of the modifiers used in filler modification on the pH of furnish slurry might be mainly attributed to the pH-buffering action, and the formation of Ca-P overgrowths on the filler surfaces as a result of addition of phosphoric acid might also contribute to the lowered system pH (Pang et al. 2003).

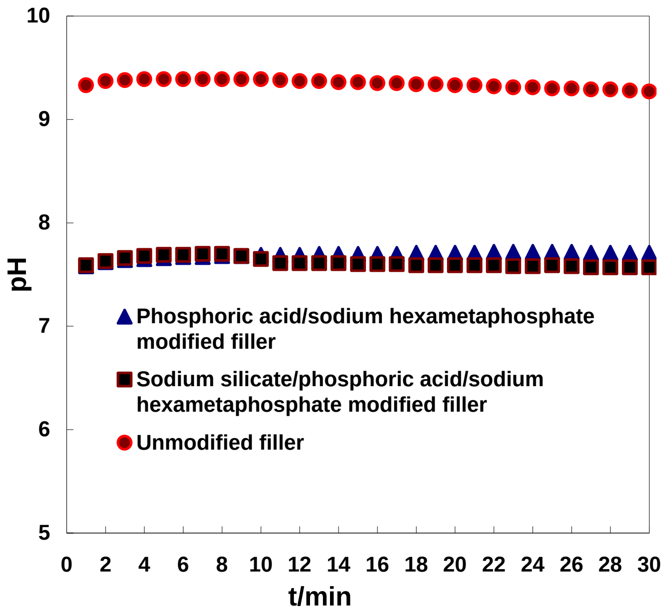


Fig. 2. Influence of filler addition on the pH profile of furnish slurry

Static Drainage Properties of the Filler-Containing Furnishes

Freeness values of the filler-containing furnishes are shown in Table 2. The freeness values of the filler-containing furnishes were all lower than that of furnish with no filler added, showing the positive effects of unmodified filler and acid-tolerant precipitated calcium carbonate fillers on furnish static drainage. Compared with the furnish containing unmodified precipitated calcium carbonate filler, the freeness values of furnishes containing acid-tolerant fillers were slightly higher. Also, these two acid-tolerant precipitated calcium carbonate fillers exhibited almost the same effects on furnish freeness. Therefore, the acid-tolerant precipitated calcium fillers could improve the static drainage property of deinked pulp derived from recycled newspaper, but their effects were slightly weaker in comparison to the unmodified precipitated calcium carbonate filler.

Table 2. Freeness Values of the Furnishes.

Sample	Freeness (°SR)
Furnish with no filler added	57.0
Furnish containing unmodified precipitated calcium carbonate filler	48.2
Furnish containing phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	52.0
Furnish containing sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	51.8

Ash Content of the Filled Papers

Ash content values of the filled handsheets are shown in Table 3. The ash content of handsheets with no filler added was as low as 4.0 wt%. For the filled handsheets, the handsheets filled with sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler gave the highest ash content, and the handsheets filled with phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler gave the lowest ash content. Thus, the retention property of sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler was better than phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler and unmodified precipitated calcium carbonate filler.

Table 3. Ash Content Values of the Filled Handsheets.

Sample	Ash content (wt%)
Handsheets without filler	4.0
Handsheets filled with unmodified precipitated calcium carbonate filler	16.0
Handsheets filled with phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	12.7
Handsheets filled with sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	16.6

Optical Properties of the Filled Papers

Optical properties of the filled handsheets are shown in Table 4. The use of unmodified precipitated calcium carbonate filler and acid-tolerant precipitated calcium carbonate fillers all strikingly improved the brightness of handsheets. The brightness of handsheets filled with acid-tolerant precipitated calcium carbonate fillers was higher as compared with the handsheets filled with the unmodified precipitated calcium carbonate filler. After modification, the brightness of handsheets increased by more than 2 %ISO, presumably indicating the alleviation of pulp darkening, achieved by enhanced acid-resistant property of the filler. For phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler, its retention in the handsheets was lower than the unmodified precipitated calcium carbonate filler (see Table 3), whereas the brightness of handsheets filled with the acid-tolerant filler was higher than that of handsheets filled with the unmodified filler, further confirming the alleviation of pulp darkening associated with the use of acid-tolerant filler. Thus, under the controlled experimental conditions, the acid-tolerant precipitated calcium carbonate fillers were more efficient in brightness improvement of the filled papers in comparison to the unmodified precipitated calcium carbonate filler.

The opacity of filled handsheets was slightly lower than that of unfilled handsheets, and the opacity of handsheets filled with phosphoric acid/sodium hexameta-phosphate modified filler was slightly higher than handsheets filled with unmodified filler.

The light scattering coefficient of filled handsheets was much higher than that of unfilled handsheets. Compared with the handsheets filled with unmodified precipitated calcium carbonate filler, the light scattering coefficient of handsheets filled with sodium silicate/phosphoric acid/sodium hexametaphosphate modified filler was slightly higher, but the light scattering coefficient of handsheets filled with phosphoric acid/sodium hexametaphosphate modified filler was much lower.

Table 4. Optical Properties of the Filled Handsheets

Sample	Brightness (%ISO)	Opacity (%)	Light scattering coefficient ($m^2 \cdot kg^{-1}$)
Handsheets without filler	61.7	97.3	52.7
Handsheets filled with unmodified precipitated calcium carbonate filler	67.8	96.4	69.0
Handsheets filled with phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	69.9	97.0	61.6
Handsheets filled with sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	70.5	95.7	69.4

Air Permeability of the Filled Papers

Air permeability values of the filled handsheets are shown in Table 5. The use of acid-tolerant precipitated calcium carbonate fillers and unmodified precipitated calcium carbonate filler strikingly increased the air permeability of handsheets. Compared with the handsheets filled with unmodified precipitated calcium carbonate filler, the air permeability of handsheets filled with acid-tolerant precipitated calcium carbonate fillers was slightly higher. Thus, the air-permeability-increasing effect of acid-tolerant precipitated calcium carbonate fillers was slightly greater than that of unmodified precipitated calcium carbonate filler.

Table 5. Air Permeability Values of the Filled Handsheets

Sample	Air permeability ($L \cdot m^{-2} \cdot s^{-1}$)
Handsheets without filler	18.5
Handsheets filled with unmodified precipitated calcium carbonate filler	28.5
Handsheets filled with phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	34.8
Handsheets filled with sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	30.1

Strength Properties of the Filled Papers

Tensile indices and burst indices of the filled handsheets are shown in Table 6. The use of modified precipitated calcium carbonate fillers and unmodified precipitated calcium carbonate filler all impaired the strength properties of filled handsheets to a certain degree. For the three fillers used in this work, the handsheets filled with sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler gave comparatively good tensile and burst strength properties. As the retention of sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler was comparatively high, filler modification with sodium silicate/phosphoric acid/sodium hexametaphosphate can possibly alleviate the negative effect of filler loading on paper strength.

Table 6. Tensile and Burst Index Values of the Filled Handsheets

Sample	Tensile index ($N \cdot m \cdot g^{-1}$)	Burst index ($KPa \cdot m^2 \cdot g^{-1}$)
Handsheets without filler	17.02	1.22
Handsheets filled with unmodified precipitated calcium carbonate filler	11.14	0.66
Handsheets filled with phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	9.93	0.59
Handsheets filled with sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler	13.28	0.65

CONCLUSIONS

1. The use of acid-tolerant precipitated calcium carbonate fillers, including phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate, and sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate, in papermaking of deinked pulp derived from recycled newspaper provided considerably more brightness improvement as compared with unmodified filler.
2. The pH values of the furnish slurries containing acid-tolerant precipitated calcium carbonate fillers were much lower than that of unmodified filler.
3. The use of acid-tolerant precipitated calcium carbonate fillers and unmodified filler all improved furnish static drainage, and the improving effect of acid-tolerant precipitated calcium carbonate fillers was slightly weaker as compared with unmodified filler.
4. Compared with the unmodified filler, the acid-tolerant precipitated calcium carbonate fillers gave slightly more increase in air permeability of the papers.
5. For tensile and burst strength of filled paper, the use of sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler gave better results as compared with the other two fillers.
6. Among the three fillers, the use of sodium silicate/phosphoric acid/sodium hexametaphosphate modified precipitated calcium carbonate filler gave the highest ash content of the filled paper, indicating the best retention performance.
7. Further work concerning the important issues such as comparisons of acid-tolerant precipitated calcium carbonate fillers with the use of carbon dioxide as a dissolution inhibitor, alkaline darkening properties of the deinked pulp, more complete statistics of the experimental data, overall characterization and evaluation of fillers, and the relevant mechanisms, is still needed in the future.

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