Lumen Loading of Bleached Pulps by In-Situ Precipitation of Filler

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ABSTRACT

The in-situ precipitation method has been used to precipitate sodium alumino-silicate as filler on the fibres using papermaker's alum and sodium silicate. The filler was produced in two ways; first in absence of fibres and second in presence of fibres i.e. in-situ loading of filler. The fresh filler produced in absence of fibre was then added to the pulp slurry. Comparison of various pulp and paper properties was made for direct loading of market filler, fresh filler loading and filler prepared in-situ with fibres. In-situ lumen loading technology provided paper with significant improvement in various properties of paper as compared to fillers directly added to the stock. Bulk and stiffness of the hand sheets prepared with in-situ precipitation were much higher than those of sheets prepared with fillers directly added to the pulp. There is no appreciable increase in brightness and whiteness of paper with in-situ precipitation. In-situ filler loaded pulps showed higher filler retention value as compared with directly filler loaded pulps.

INTRODUCTION

For some applications opacity, strength, stiffness and bulk of paper become the critical properties. Using more filler in paper can increase opacity, but this usually decreases paper strength and stiffness. Thus, paper structure should be altered in such a way that desirable paper properties are maintained. Both pigments and cellulosic materials are being used as effectively as possible to maintain the properties mentioned above. New developments such as precipitated fillers and pigments are known to offer new opportunities to improve paper properties. Improved interaction of precipitated pigments with fibres and fines may provide papermakers with new ways to improve paper properties. It has also been assumed that inorganic salts precipitated within fibre walls may increase fibre stiffness with positive impact on light scattering and paper stiffness (1).

Fibre-loading technology may offer a new route to improve strength of paper, formation and retention simultaneously, since filler retention may be decreasingly dependent upon chemical retention, flocculation, and agglomeration. The studies on filler-loading shows that the filler can be incorporated with/within fibre by three different ways: i) the direct loading of filler with fibres - presently used in most of the pulp and paper industries, ii) lumen loading of softwood fibres by mechanical diffusion of filler inside fibre lumen through pit apertures, and iii) lumen and/or cell wall loading by in-situ precipitation of filler. The last technique has been used to incorporate the filler inside fibre lumen by chemical precipitation.

Methods for incorporating fillers inside fibres have been extensively studied, not only for better filler retention but also for better paper properties (2-5). Many investigators reported fibre wall filled paper to exhibit greater tensile, burst, and tear strength than corresponding conventional paper. Better strength obtained is assumed to be due to increased hydrogen bonding of fibres, because the filler is located inside the fibres (2,4,6). In precipitation, the approach has usually been to saturate pulp fibres with a soluble salt and to precipitate filler by another soluble salt. The information available on in-situ precipitation of filler inside hardwood fibres is limited (7). The present study focuses on improving the properties of paper by structuring precipitated filler and pulp material in a new way.

EXPERIMENTAL

Materials

The pulp used in this study was never dried pulp obtained from an integrated pulp & paper mill in north India using hardwoods, mainly 50% Eucalyptus and 50% Poplar. The beating of pulp was done in the PFI mill (Hamjern Maskin A/S, Hamar, Norway; PFI mill no. 616) to achieve 420 CSF value. Fresh sodium alumino-silicate (SAS) was prepared in two different ways, i) precipitation in absence of fibres, and ii) in-situ precipitation (in presence of fibres). Commercial grade sodium silicate and papermaker's alum were collected from indigenous sources.

Precipitation of sodium alumino-silicate (SAS)

The process used for in-situ precipitation of SAS (i.e. precipitation in presence of fibres, SAS-ISP) is as follows. A fixed amount of 44% alum solution was impregnated into the never dried beaten pulp. The mixing of this slurry was done for about 10 minutes in high-speed disintegrator so that the lumen became filled with the ionic solution. Thereafter, sodium silicate of 35% concentration was added gradually at a fixed addition rate for a particular time period in the impregnated pulp slurry. The slurry was further agitated for 10 minutes to achieve the saturation point. To know the actual amount of filler precipitated within lumen, the pulp was washed in Bauer McNett Classifier to remove the excess filler not attached or attached loosely to the outer surfaces of the fibres. The general process steps involved in this process are depicted in Figure 1. Sodium alumino-silicate in absence of fibres was also prepared in the similar manner as described above.

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Direct filler loading

The directly added fillers were Hydrex-P (sodium magnesium alumino silicate) and Finex (magnesium silicate) in the form of slurry having 20% solids. The freshly prepared sodium alumino-silicate (SAS) was also added directly in the pulp as other fillers. The retention aid (Percol-47) was used with Hydrex-P and freshly prepared SAS to achieve good first pass ash retention. Paper hand sheets were prepared at 2.5, 5.0 and 7.5% ash levels with all fillers.

Sheet forming

The paper hand sheets having a basis weight of 70 g/m^2 were prepared for all the experiments. In case of SAS-ISP, sheets were prepared without doing classification of pulp (as such precipitated filler and fibres). This slurry consisted both lumen loaded fibres as well as filler outside fibres. The properties of hand sheets formed by in-situ precipitated sodium alumino-silicate (SAS-ISP) were compared to those of directly added fillers i.e., freshly prepared sodium alumino-silicate (SAS), Hydrex-P and Finex.

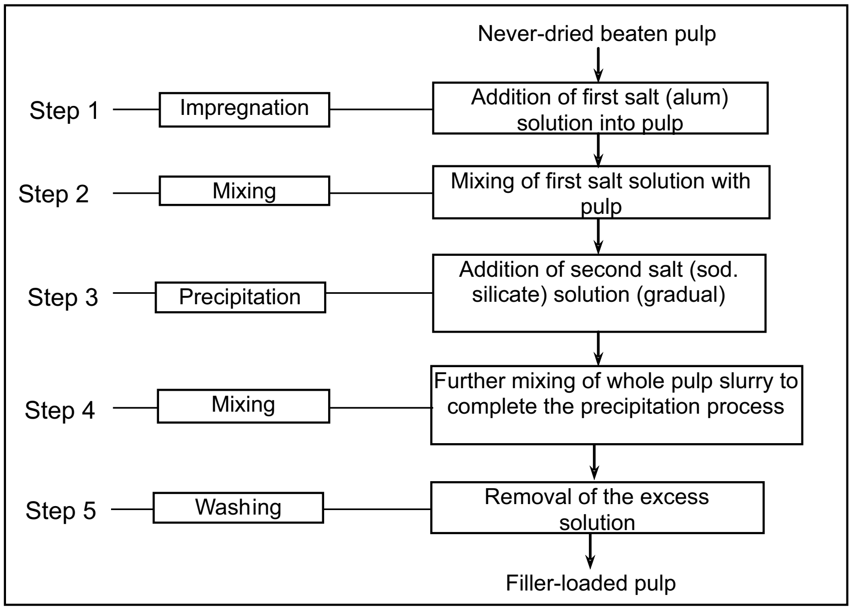

Fig. 1: General process steps involved in SAS-ISP

Table 1: Properties of different fillers

Parameter	Finex	Hydrex-P	SAS
Brightness (%ISO)	93.5	99.2	96.3
CIE Whiteness	94.2	101.7	94.9
Particles < 2 μm (%)	20.0	78.0	87.3
pH	9.21	9.70	7.22

Table 2: Extent of SAS precipitation outside and inside fibers

Sr. no.	Ash in pulp (%)	
	Before washing	After washing in Bauer McNett classifier
1	4.0	1.8
2	5.5	2.1
3	7.1	2.6
4	10.1	3.0

Analytical Techniques

The physical properties of the hand sheets were determined according to relevant standard methods. The optical properties were tested on Datacolor (Datacolor, USA; model: Spectraflash 300 UV) brightness tester. Ash in paper was estimated according to Tappi Test Method T-211 om-93 by the incineration of paper at 575±25 °C in muffle furnace. First pass ash retention was calculated from the ratio of ash in paper and ash in pulp. Particle size of filler was determined using 10% slurry in Particle Size Analyzer (Microscan II from Quantachrome Corporation, USA).

RESULTS AND DISCUSSION

The properties of different fillers are reported in Table 1, which shows that brightness and whiteness of Hydrex-P is the maximum, whereas SAS is the finest filler having around 87.3% particle less that 2 micron size.

To determine the actual amount of SAS precipitated within the fibre, the pulp was washed in Bauer McNett classifier after the complete process of in-situ precipitation of filler. During the washing, the fines were removed which also carried away the SAS precipitated outside the fibre. The total ash in the pulp decreased on washing. This trend was seen at all the ash levels (Table 2). The ash content in the washed pulp may be assumed to represent the amount of SAS precipitated inside the fibres. It can be seen from Table 2 that the amount of SAS precipitate inside the fibre does not increase in the same proportion as the total SAS precipitate (ash content) increases; after certain level, precipitation takes place mainly at the outer surface of the fibre.

First pass ash retention (FPAR) of SAS-ISP was the maximum. It was due to the

Table 3: Effect of different fillers on

Parameter	Blank	Finex	Hydrex-P	SAS	SAS-ISP
(a) At 2.5% ash level					
Ash in hand sheet (%)	0.4	2.4	2.4	2.6	2.5
FPAR (%)	-	53.3	55.8	55.3	67.6
Bulk (cc/g)	1.27	1.26	1.28	1.29	1.31
(b) At 5% ash level					
Ash in hand sheet (%)	0.4	5.0	5.1	5.0	4.9
FPAR (%)	-	54.9	52.0	51.0	69.0
Bulk (cc/g)	1.27	1.25	1.31	1.30	1.36
(c) At 7.5% ash level					
Ash in hand sheet (%)	0.4	7.4	7.5	7.5	7.5
FPAR (%)	-	56.9	40.5	39.7	72.1
Bulk (cc/g)	1.27	1.25	1.32	1.30	1.42

Table 4: Effect of different fillers on

Parameter	Blank	Finex	Hydrex-P	SAS	SAS-ISP
(a) At 2.5% ash level					
Ash in hand sheet (%)	0.4	2.4	2.4	2.6	2.5
Breaking length (m)	6138	5409	5309	5355	6088
Burst index (kN/g)	4.20	3.96	3.26	3.40	4.02
Tear index (mNm^2/g)	8.12	8.07	8.11	8.06	8.16
Bending stiffness (mNm)	1.16	1.08	0.96	0.98	1.22
Scott Bond (J/m^2)	582	535	498	514	622
(b) At 5% ash level					
Ash in hand sheet (%)	0.4	5.0	5.1	5.0	4.9
Breaking length (m)	6138	5068	4917	5001	5402
Burst index (kN/g)	4.20	3.60	3.01	3.15	3.74
Tear index (mNm^2/g)	8.12	7.96	7.76	7.78	8.31
Double fold (number)	134	47	27	25	116
Bending stiffness (mNm)	1.16	1.02	0.94	0.82	1.13
Scott Bond (J/m^2)	582	520	376	458	620
(c) At 7.5% ash level					
Ash in hand sheet (%)	0.4	7.4	7.5	7.5	7.5
Breaking length (m)	6138	4626	4554	4667	5116
Burst index (kN/g)	4.20	3.23	2.65	2.80	3.47
Tear index (mNm^2/g)	8.12	7.84	7.14	7.59	8.55
Double fold (number)	134	33	10	19	85
Bending stiffness (mNm)	1.16	0.95	0.92	0.75	1.03
Scott Bond (J/m^2)	582	513	272	379	620

reason that some amount of the filler was precipitated inside the fibres. The same trend can be seen at all the ash levels (Table 3). The FPAR of Finex was also good because of its larger particle size. The higher FPAR value shows cleaner white water system. Therefore, in case of SAS-ISP, the white water system will be cleaner in comparison to the white water system of direct filler loaded pulps. Similar results on better filler retention in case of in-situ precipitation have been reported earlier also (6,7).

Normally, with direct filler loading, the bulk of paper sheet decreases with increase in ash content. From Table 3, it can be seen that the bulk is also dependent upon the type of filler used. In some cases, it decreases whereas in others it increases. The bulk of paper decreased in case of Finex, whereas in case of Hydrex-P and SAS it increased as compared with the blank. This difference in behavior may be due to the bulk density of fillers. The bulk in case of SAS-ISP increased appreciably probably due to some precipitation of filler inside the fibres, which decreased the collapsivity of fibres, resulting in increase in thickness of the paper. From these results, it is further confirmed that some of the filler has been precipitated within the fibre (lumen) in case of SAS-ISP.

Stiffness of paper depends upon the type of fibres and thickness of paper. With increase in thickness, stiffness increases. It can be seen from Table 4 that the stiffness of paper hand sheets was the maximum in case of SAS-ISP as compared to that of direct filler loaded sheets. It may be due to rather higher bulk of SAS-ISP. The stiffness normally decreases with increase in filler loading. However, in case of SAS-ISP, initially the stiffness increased and afterwards decreased with increase in ash level but to a smaller extent as compared to other fillers (Tables 4).

All the physical strength properties decreased to a large extent with direct loading of fillers whereas in case of SAS-ISP, all the strength properties were on the higher side as compared with direct filler loading. The breaking length decreased with the addition of filler in all the cases (Table 4). The extent of drop in breaking length has been much lower in case of SAS-ISP as compared with direct filler loading which is in agreement with the earlier findings of Siven and Manner (7).

For a given filler type, the smallest filler have the most detrimental effect on paper strength. A good indication of the role of particle size was that the loss of burst index increased with the total surface area of fillers (Li *et al.,* 2002). From Tables 4, it can be seen that burst index of paper hand sheets decreased with increase in ash level in all the cases. However, the loss in burst index was much lower in case of SAS-ISP as compared with direct filler loading. The highest loss was seen in case of Hydrex-P, followed by SAS and Finex. The effect of particle size can easily be seen in all the cases.

In case of direct filler loading, the tear index of paper hand sheets decreased with addition of filler (Tables 4). The trend of decrease in tear index was different for different fillers. It was highest in case of Hydrex-P, followed by SAS and Finex, whereas in case of SAS-ISP, this trend was opposite. There was an increase in tear index with increase in ash content. This increase may be because of filling of lumen with

Table 5: Effect of filler on optical properties

Parameter	Blank	Finex	Hydrex-P	SAS	SAS-ISP
(a) At 2.5% ash level					
Ash in hand sheet (%)	0.4	2.4	2.4	2.6	2.5
Brightness (%ISO)	80.8	81.4	83.0	82.4	80.9
CIE Whiteness	62.1	62.8	67.0	66.2	62.4
Opacity (%)	75.9	76.3	80.5	78.9	76.0
Scattering coefficient (m^2/kg)	27.4	28.1	35.4	31.9	27.5
(b) At 5% ash level					
Ash in hand sheet (%)	0.4	5.0	5.1	5.0	4.9
Brightness (%ISO)	80.8	81.6	84.5	83.9	81.2
CIE Whiteness	62.1	63.3	69.7	69.4	64.1
Opacity (%)	75.9	76.4	82.0	80.7	76.1
Scattering coefficient (m^2/kg)	27.4	28.4	41.1	35.6	27.8
(c) At 7.5% ash level					
Ash in hand sheet (%)	0.4	7.4	7.5	7.5	7.5
Brightness (%ISO)	80.8	82.0	85.3	84.5	81.8
CIE Whiteness	62.1	63.6	71.5	70.6	66.4
Opacity (%)	75.9	77.0	82.8	81.2	76.3
Scattering coefficient (m^2/kg)	27.4	29.3	41.8	37.3	28.0

SAS, so that the force required to tear the sheet increased with increase in effective fibre thickness.

The internal bond strength (Scott bond) of paper hand sheets prepared with direct filler loaded pulp in comparison to blank decreased with increase in ash level, whereas it slightly increased in case of SAS-ISP (Table 4). The internal bond strength depends upon the thickness and internal bonding of fibres. If the internal bond strength of a paper increases, it means either one or both of the thickness and internal bonding of fibre have increased. This was again a sign of proof for SAS-ISP that an appreciable part of the filler was precipitated inside the fibre lumen. Therefore, the fibre bonding was better and thickness was more in this case. It can be seen that in case of SAS-ISP, Scott bond increased with increase in ash level, whereas it decreased in case of direct filler loading. The highest decrease in Scott bond was with Hydrex-P, followed by SAS and Finex.

All the optical properties were on the lower side in case of SAS-ISP as compared with direct filler loading (Table 5). In fact, a major portion of SAS-ISP was precipitated inside the fibres and it was not affecting the reflectance of light in the similar manner as direct SAS addition. In case of direct filler loading, the filler was interacting directly with the light hence there was more reflection of light. The highest brightness was achieved in case of sheets prepared with Hydrex-P because of its high brightness (Tables 5 and 1). The freshly prepared SAS was also having good brightness as compared with other fillers. When the comparison is made between SAS and SAS-ISP (i.e. as such filler preparation in absence of fibres and in-situ precipitation of filler in presence of fibres respectively), it was seen that the brightness of paper in case of SAS-ISP was quite low. Opacity is also quite low in case of SAS-ISP as compared with direct filler loading. These results further confirm that by in-situ precipitation of SAS, appreciable proportion of the filler gets precipitated inside the fibre, which is not affecting the light absorption.

CONCLUSIONS

The in-situ precipitation method has been used to precipitate sodium alumino-silicate as filler inside the fibres. The following conclusions can be drawn from this study:

1. The in-situ precipitation of sodium alumino-silicate in hardwood fibres helps in producing paper with greater stiffness and bulk than the paper with the fillers directly added to the pulp.

2. All the strength properties are higher in case of SAS-ISP as compared to those of direct filler loading.

3. Filler retention for in-situ filler loading is higher than the retention for direct filler loading. In most conventional fillers, a high dose of retention aid is needed except with talc.

4. There is no appreciable increase in brightness and whiteness of paper with SAS-ISP as the filler gets precipitated inside the fibres.

5. Overall, the in-situ precipitation is advantageous in improving/ maintaining some of the important properties of paper like bulk and stiffness, which normally decrease on conventional filler loading. However, it is advisable to restrict the ash content inside the fibre to a low level, say 2.0-2.5% (which is sufficient to get the benefits of bulk, stiffness and other strength properties) as it may be difficult and expensive to increase the ash inside the fibre further. Ash content of the paper can be subsequently increased to the desired level by conventional filler loading while getting the benefits of ISP.

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