

The influence of impulse drying on the performances of papers containing cationic starch and calcium carbonate

PAULA MENDES, HELENA AGUILAR, JOÃO RANITA, MOHAMED NACEUR BELGACEM,
JACQUES SILVY, AND CARLOS A.V. COSTA

ABSTRACT: Impulse-dried sheets made with bleached kraft pulps beaten at 25°SR and containing 2% cationic starch showed excellent structural consolidation. In fact, all the mechanical and structural properties except stiffness were enhanced. During impulse pressing, cationic starch handsheets from pulps beaten at 25°SR exhibited less delamination than those made from pulps beaten at 40°SR. Web sticking (to the heated platen press surface) was observed for sheets filled with precipitated calcium carbonate (15% CaCO₃). This sticking was attributed to the presence of calcium carbonate, which makes the paper sheets have smaller void spaces, leading to the delamination of the paper web.

Application: Calcium carbonate may eventually be a suitable filler for papers dried by impulse drying, but first there are some problems that will have to be solved.

Impulse drying is a new consolidation technique that combines the mechanical compression aspects of the wet web with an intense hot surface drying [1]. The results of work over the past 20 years have indicated that very high drying rates and some improvements in sheet density could be achieved in additive-free papers of various furnish compositions [2, 3]. Unfortunately, in some cases the steam pressure at the corresponding temperature was too high, and the sheet ruptured or delaminated as it left the nip [4–6]. It was believed that this phenomenon could be avoided if the pressing operation could be properly controlled [7]. Although impulse drying was shown to be capable of producing smoother and stronger paper sheets, some properties, such as stiffness and opacity, were impaired.

In papermaking, the application of fillers is particularly important when opacity and/or printability are required, as in the case of copy paper grades [8]. Moreover, the difference in prices between bleached pulp and fillers has led papermakers to substitute cheaper mineral filler for the more expensive fiber, which has allowed them to save costs [9]. Over the past decade, a major trend toward manufacturing printing and writing papers under alkaline conditions has pointed to a significant growth in the use of calcium carbonate [10]. Besides the properties mentioned, this inorganic filler

also imparts to papers low permeability and high sheet density, but it has also decreased strength properties [11, 12]. Today, the preferred system for reducing the strength loss caused by adding filler involves the use of cationic starch as a dry strength agent [13].

Cationic starch is one of the most important additives used in paper manufacturing to increase the dry strength, the retention, and the dewatering rate [14]. In fact, cationic starch creates new bridges among fibers. The free hydroxyl groups of starch participate in hydrogen bonding at the surfaces of cellulose fibers, which are more pervasive points of attachment than those resulting from fiber-fiber bonds [15]. Moreover, cationic starch increases the specific bond strength that is directly related to inter-fiber bonding [16].

To the best of our knowledge, there is no report dealing with studies on impulse drying and the influence of additives. Therefore, we decided to study two common additives used in copy paper grades. We conducted laboratory-scale experiments on paper sheets made from bleached kraft pulps from *Eucalyptus globulus* beaten at two refining levels and containing either cationic starch or calcium carbonate. We analyzed the impact that impulse drying has on the structural, mechanical, and optical properties of the hot-pressed paper samples.

EXPERIMENTAL PROCEDURES

Materials

The pulp used in this work was a bleached kraft pulp from *E. globulus*. Hardwood pulp was chosen because it usually provides 80% of the fiber for printing and writing grades. The cationic starch used was made from potato derivatives modified by alkyl chains bearing quaternary ammonium groups at a substitution degree of 0.034. We chose this degree of substitution based on previous research dealing with the effect of cationic starch on paper mechanical properties [17].

Precipitated calcium carbonate (CaCO₃) was used as filler. Suppliers gave the specific surface area as 4.2–4.8 m²/g (BET surface area) and the average particle size as 2.75 µm. The retention aid used for filled paper was a commercial polyethyleneimine polymer (Polymine SK) supplied by BASF. We used deionized water in all experiments.

METHODS

Preparation of pulp suspension

The pulp was soaked in water for 24 h. After disintegration for 20 min, the pulp was beaten in a Valley beater to either 25°SR or 40°SR (Schopper-Riegler). The homogeneous fiber suspension was diluted to a concentration of 0.8% (i.e., 8 g of oven-dried fibers per liter of water).

Preparation and addition of cationic starch to the furnish

A solution of cationic starch having 5% mass concentration was prepared and gelatinized by boiling for 30 min with gentle agitation. The solution was diluted at 1% (w/w), and 2% cationic starch (w/w on o.d. pulp) was added to the fiber suspension. The mixture was left to reach adsorption equilibrium (about 20 min). Paper handsheets were prepared from this furnish.

Addition of calcium carbonate to the furnish

Under constant stirring (900 rpm), we added calcium carbonate to the 0.8 g/L fiber suspension (15% CaCO₃). When the furnish was homogeneous, we added Polymin SK (0.5% w/w on o.d. pulp) as a retention aid. The suspension was maintained under constant stirring at 900 rpm.

Sheet forming

We used a standard sheet former to prepare handsheets at a basis weight of 80 g/m². The diameter of the sheets was 16.5 cm. Since the prototype used to press samples at different temperatures has a smaller area (7.5 cm by 7.5 cm), we divided the initial sheet into two samples with the desired size. The plastic frame we used to divide the sample is placed onto the surface of the forming wire before the pulp is introduced. This process does not disturb the sheet formation, and it simplifies the cutting of the wet samples, which would otherwise be a difficult task.

Pressing and drying methods

We carried out impulse drying experiments on a platen hydraulic press. On this press, the paper web remains stationary as it is pressed between the two plates. The stainless steel plate on top is solid and very smooth, and it is heated by four electrical cartridge heaters.

Beneath the paper web is a felt, under which is a sintered stainless steel lamina with pores about 200 µm in diameter. The plate that holds this lamina has several parallel grooves that take water and air away from the paper. This sintered metal plate also confers stability and homogeneity to the system.

In our experiments, we used the following conditions:

- Web dryness prior to impulse drying: 30%–40%
- Pressing temperatures: 25°C, 80°C,

		100% <i>Eucalyptus globulus</i>						
		25°SR				40°SR		
		25°C	150°C	200°C	250°C	150°C	200°C	250°C
Structural properties	Basis weight (g/m ²)	83.0	79.9	79.9	85.2	83.3	82.1	79.8
	CV (%)	3.4	2.6	2.4	1.4	2.3	2.7	2.8
	Thickness (µm)	147	121	110	121	126	124	129
	CV (%)	3	5	5	5	6	6	16
	Density (g/cm ³)	0.57	0.66	0.73	0.70	0.66	0.66	0.62
	PAFNOR index (µm/Pa.s)	40.0	22.4	13.7	11.0	3.6	3.3	3.8
Mechanical properties	CV (%)	6.3	9.4	15.3	9.3	4.7	17.4	24.8
	Roughness Bendtsen's method (ml/min)	345	241	253	227	315	265	297
	CV (%)	8	14	10	19	22	17	35
	Smoothness Bekk's method (s)	76.0	186.0	168.8	166.3	73.1	77.3	69.6
	CV (%)	11.6	14.1	9.9	18.9	31.3	31.2	58.7
	Tensile index (N.m/g)	27.8	34.1	37.7	44.9	48.8	52.5	63.4
Optical properties	CV (%)	10.1	13.1	14.5	4.6	18.3	9.4	11.3
	Elongation at break (%)	2.9	3.1	3.2	4.5	7.0	8.6	7.3
	CV (%)	27.8	21.4	24.7	9.4	15.1	18.2	11.3
	Young's modulus (MPa)	1359	1740	2102	1425	1368	1385	1426
	CV (%)	4	14	11	12	12	20	21
	Stiffness (mN.m)	0.519	0.377	0.297	0.303	0.396	0.398	0.230
Optical properties	CV (%)	11.062	13.062	4.367	12.678	13.221	9.022	6.267
	Internal Cohesion strength (J/m ²)	235	231	334	470	431	491	441
	CV (%)	7	12	8	5	24	9	8
	Burst index (kPa.m ² /g)	1.69	2.29	2.63	2.23	3.76	4.10	3.22
	CV (%)	18.90	22.04	4.12	10.53	6.64	6.86	5.95
	Tear index (mN.m ² /g)	5.2	5.2	6.0	6.9	7.4	9.5	10.9
Optical properties	CV (%)	0.0	0.0	0.0	7.4	2.8	2.8	7.4
	Brightness (%)	87.4	86.4	85.0	84.6	84.7	83.9	83.1
	CV (%)	0.1	0.1	0.4	0.6	0.1	0.5	0.2
	Opacity (%)	78.5	75.7	72.7	73.8	75.6	75.3	76.2
	CV (%)	0.7	2.0	2.3	1.7	0.4	2.0	2.7
	Light scattering coefficient (m ² /kg)	43.41	35.81	31.22	33.45	34.31	32.97	35.95
Optical properties	CV (%)	2.47	1.43	7.23	5.73	1.14	5.67	8.32
	Light absorption coefficient (m ² /kg)	0.18	0.20	0.22	0.20	0.29	0.33	0.27
	CV (%)	4.02	2.69	3.99	6.04	2.40	11.71	7.58
	Yellow index	4.49	4.18	4.48	5.90	3.77	3.72	6.41
	CV (%)	1.40	1.65	4.71	5.49	1.64	2.49	1.67

I. Physical properties of impulse-pressed paper having a basis weight of 80 g/m².

150°C, 200°C, 250°C, and 350°C

- Dwell time: 50 ms
- Peak pressure: 6.2 MPa
- Initial felt moisture content: about 15%.

After pressing, the samples were dried under restricted shrinkage at 105°C for 10 min.

Handsheet characterization

Impulse-dried samples were conditioned for 24 h at 23°C and 50% RH before testing. Tests carried out for structural properties included basis weight, thickness, air permeability, roughness (Bendtsen's method), and smoothness (Bekk's method). Tests for mechanical properties included tensile strength, elongation at rupture, Young's modulus, stiffness, internal cohesion strength, burst strength, and tear strength. We tested optical properties for brightness, opacity, light scattering coefficient, light absorption coefficient, and yellow index.

All the data obtained for the structural properties were determined as the arithmetic mean of 10 measurements, except for thickness, which was based on 20 values. The results obtained for the mechanical and optical properties are averages of five measurements, except for the burst strength, which was an average of three measurements.

The retention of filler was evaluated by an incineration method according to TAPPI 413 om-85.

RESULTS AND DISCUSSION

The structural, mechanical and optical properties of the free-additive paper samples, and those either with 2% of cationic starch or 15% of calcium carbonate are summarised in **Tables I and II**, respectively. In pressing, the paper sheets with cationic starch had a problem with sticking. The paper samples stuck to the top plate of the press, making it impossible

		2% Cationic Starch			15% CaCO ₃					
		25°SR			25°SR			40°SR		
		25°C	150°C	200°C	25°C	200°C	250°C	150°C	200°C	250°C
Structural properties	Basis weight (g/m ²)	87.0	78.8	91.7	83.6	85.9	84.9	86.1	80.4	81.6
	CV (%)	4.9	2.9	1.2	2.4	1.3	3.2	1.3	3.0	4.3
	Thickness (μm)	167	118	124	139	132	128	139	141	144
	CV (%)	5	4	4	4	6	5	4	8	15
	Density (g/cm ³)	0.52	0.67	0.74	0.60	0.65	0.66	0.62	0.57	0.57
	P _{AFNOR} index (μm/Pa.s)	82.1	13.5	14.0	12.5	29.5	26.5	22.5	11.5	9.8
	CV (%)	3.0	10.8	13.8	13.2	5.9	17.2	12.5	17.5	30.0
Roughness Bendsen's method (ml/min)	508	224	235	291	350	350	299	554	546	
	CV (%)	13	5	4	13	12	11	15	22	38
Smoothness Bekk's method (s)	37.6	193.6	150.7	97.4	80.2	82.6	111.9	27.8	35.2	
	CV (%)	10.4	2.2	22.0	15.6	18.4	16.9	15.4	27.6	42.9
Mechanical properties	Tensile index (N.m/g)	34.2	48.3	44.1	53.6	34.5	42.9	41.9	37.4	42.2
	CV (%)	6.9	17.8	25.7	2.8	5.2	4.5	13.1	3.5	19.0
	Elongation at break (%)	3.5	5.9	6.6	9.4	3.3	4.0	5.7	6.1	6.3
	CV (%)	9.6	36.5	39.6	11.0	6.3	8.7	20.8	20.8	29.1
	Young's modulus (MPa)	1143	1466	1525	983	982	1278	951	799	811
	CV (%)	13	12	16	13	14	12	22	23	18
	Stiffness (mN.m)	0.542	0.330	0.414	0.358	0.200	0.211	0.245	0.202	0.228
	CV (%)	16.156	15.669	6.980	20.294	23.481	35.686	29.252	19.528	45.641
Internal Cohesion strength (J/m ²)	281	485	404	273	222	234	320	Almost complete delamination	Almost complete delamination	
	CV (%)	6	14	15	11	29	34			20
Burst index (kPa.m ² /g)	2.30	4.36	4.18	3.95	2.01	2.12	2.44	2.18	1.89	
	CV (%)	17.32	8.90	12.87	8.44	26.64	0.79	19.49	4.04	37.80
Tear index (mN.m ² /g)	6.6	9.1	8.4	8.2	6.0	5.4	7.4	7.6	7.3	
	CV (%)	9.3	9.3	9.3	3.5	3.5	0.0	11.8	11.8	3.0
Optical properties	Brightness (%)	78.5	85.3	86.7	75.9	84.9	81.3	80.3	77.1	75.1
	CV (%)	3.0	0.1	0.1	1.3	5.5	1.0	2.2	0.6	0.5
	Opacity (%)	84.8	76.1	73.9	87.7	82.5	82.7	84.9	86.7	86.1
	CV (%)	2.2	0.7	0.9	1.1	1.2	0.8	1.0	1.0	1.0
	Light scattering coefficient (m ² /kg)	43.66	37.01	34.70	54.52	49.44	48.73	52.90	53.48	49.01
	CV (%)	8.08	2.27	2.74	3.36	4.94	3.41	6.17	5.27	5.31
Light absorption coefficient (m ² /kg)	0.63	0.22	0.17	0.80	0.31	0.35	0.45	0.67	0.76	
	CV (%)	45.41	3.62	3.34	14.55	6.16	5.82	14.29	4.49	4.30
Yellow index	6.26	5.12	4.61	10.39	7.57	8.64	8.86	10.01	10.84	
	CV (%)	19.93	1.44	1.15	2.85	1.40	7.85	13.41	2.14	2.24

II. Physical properties of impulse-pressed paper having a basis weight of 80 g/m² and either 2% cationic starch or 15% CaCO₃.

for us to recover the sheet after pressing. For the higher degree of beating (40°SR), the cationic starch sheets presented sticking at 80°C and 200°C, and almost complete delamination was observed in sheets pressed at 150°C, 250°C, and 350°C. Therefore, we were not able to characterize this set of papers. As for the lower degree of beating (25°SR), the delamination phenomenon was observed only in the cationic starch paper sheets pressed at 250°C and 350°C, but the paper samples pressed at 80°C stuck to the platen press.

The different behavior exhibited for the two beating degrees could be explained by the decrease in air permeability with increasing beating. Since the air permeability is lower, the rate at which liquid is eliminated from the sheet is also lower because water is unable to

flow easily toward the felt surface. In cationic starch sheets that do not delaminate or stick, the impulse drying technique enhances the web consolidation, which results in an increase in density, as Fig. 1 shows. This increase in density provokes a decrease in air permeability for the impulse-pressed sheets.

In this work we defined the variation as the percent gain or loss of a given parameter with respect to that of samples pressed at room temperature. The air permeability is higher in cationic starch sheets pressed at room temperature than for additive-free sheets pressed at the same temperature. These expected results could be attributed to the flocculation that cationic starch induces in the paper sheet.

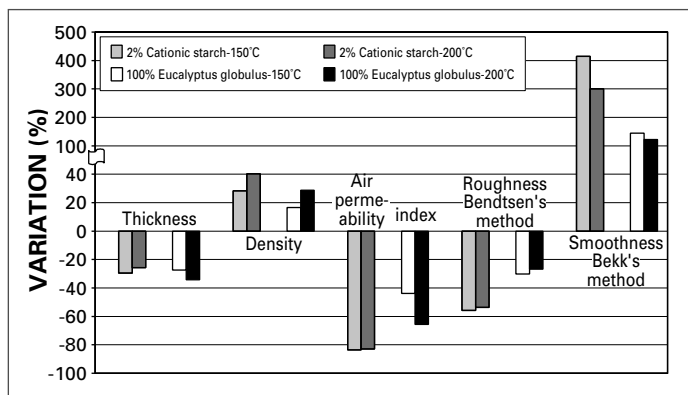
The smoothness of the paper surface is an important parameter, particularly

for printing papers. Impulse drying produces sheet surfaces that are highly closed, which means a high degree of smoothness, as shown in Fig. 1. An increase in the pressing temperature increases most of the mechanical properties, as represented in Fig. 2. This increase was more pronounced for paper sheets with cationic starch than for additive-free sheets.

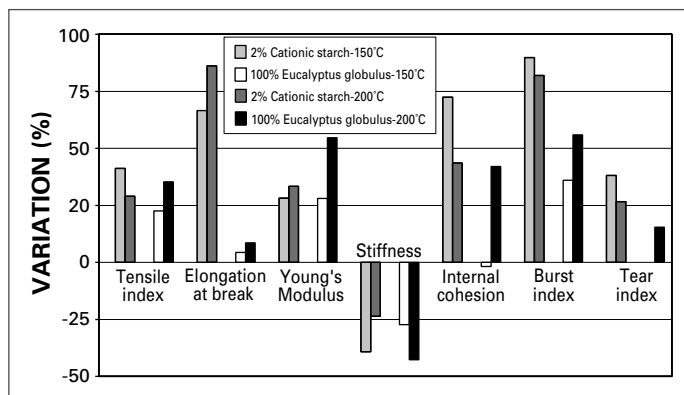
Because press drying made the web more dense, it led to good paper bonding and consequently increased the strength-to-rupture of the samples. Figure 2 gives the variation obtained for elongation at break. Higher elongation values were attained with the impulse drying technique, which positively affects the way paper withstands dynamic impacts. Moreover, this property has a beneficial effect on other mechanical properties such as tearing and burst strengths. The Young's modulus (apparent modulus of elasticity) was enhanced with increasing impulse drying temperature. The increase of fiber bonding improved the stress distribution and diminished the creep in the sheet structure, which led to the increase in Young's modulus.

The stiffness of the paper pressed at 150°C and 200°C was lower than for paper sheets pressed at room temperature. Stiffness is proportional to the in-plane elastic modulus and to the third power of the sheet thickness for a constant value of the sheet density. Thus, although the increase of the pressing temperature enhanced the in-plane Young's modulus, it also decreased the thickness, resulting in an overall decrease of paper stiffness. As shown in Fig. 3, brightness is slightly improved by impulse drying, and yellow index was reduced for paper containing cationic starch. Because the opacity is dependent on both parameters, light scattering and light absorption coefficients, the opacity was reduced by 14% for cationic starch paper samples pressed at 200°C.

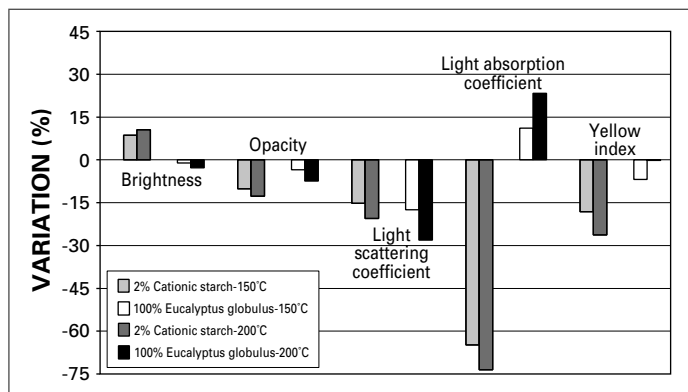
The retention of CaCO₃ into the tested sheets was found to be 82% and 83% for 25°SR and 40°SR, respectively. Paper sheets containing calcium carbonate had problems with delamination and sticking. There are two possible explanations. One explanation pertains to the lower porosity of the paper sheets containing calcium carbonate; the second pertains to the smaller number of bonds between



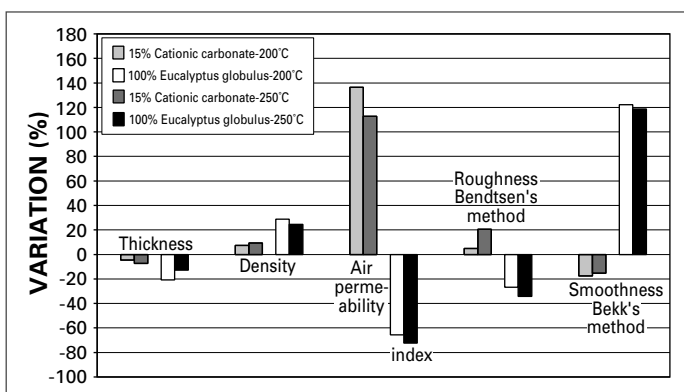
1. Variation in structural properties of impulse-dried samples pressed at 150°C and 200°C with respect to samples pressed at 25°C for additive-free sheets and for sheets with 2% cationic starch (25°SR).



2. Variation in mechanical properties of impulse-dried samples pressed at 150°C and 200°C with respect to samples pressed at 25°C for additive-free sheets and for sheets with 2% cationic starch (25°SR).



3. Variation in optical properties of impulse-dried samples pressed at 150° and 200°C with respect to samples pressed at 25°C for additive-free sheets and for sheets with 2% cationic starch (25°SR).



4. Variation in structural properties of impulse-dried samples pressed at 200°C and 250°C with respect to samples pressed at 25°C for additive-free sheets and sheets with 15% CaCO₃ (25°SR).

fibers as a result of the filler. Paper samples from fibers beaten to 25°SR and pressed at 80°C and 150°C stuck to the press plate. The same behavior was observed for sheets made from 40°SR pulp and pressed at 25°C and 80°C. In addition, paper samples made from both 25°SR and 40°SR pulps but pressed at 350°C presented complete delamination, with the sheet practically splitting apart.

In these samples, no measurements could be made. Although we can characterize the paper sheets made of 25°SR pulp and pressed at 25°C, 200°C, and 250°C, as well as the paper sheets made of the 40°SR pulp and pressed at 150°C, 200°C, and 250°C, only the 25°SR sheets pressed at 25°C presented an appropriate appearance. In fact, for the sheets beaten at 25°SR and pressed at 200°C and 250°C, large and small bubbles were detected on the felt side surface of the sheet. For sheets made from fibers beaten to 40°SR, the visual appearance is worse than that observed for sheets made from pulps beaten at 25°SR. In fact, the three sets of 40°SR samples pressed at 150°C, 200°C, and 250°C exhibited a clear tendency to

delaminate. Paper samples pressed at 150°C presented small blisters on the felt side, whereas the samples pressed at 200°C and 250°C exhibited large bubbles on the press side surface. Internal cohesion, which reflects the presence of some modes of delamination, decreased for higher temperatures. These results confirm the presence of delamination in these paper sheets. It was impossible to measure the internal cohesion of paper sheets beaten at 40°SR and pressed at 200°C and 250°C because of the almost complete delamination of the paper samples.

Figures 4, 5, and 6 present some of the physical properties of additive-free sheets and those with 15% calcium carbonate from pulp refined at 25°SR. The increase in density is more pronounced in sheets without additives than in those containing 15% calcium carbonate (Fig. 4). In paper containing CaCO₃, although thickness decreased and density increased, the other structural properties were affected negatively. Most of the mechanical properties also decreased with increasing pressing temperature for this set of papers. Therefore, the physical

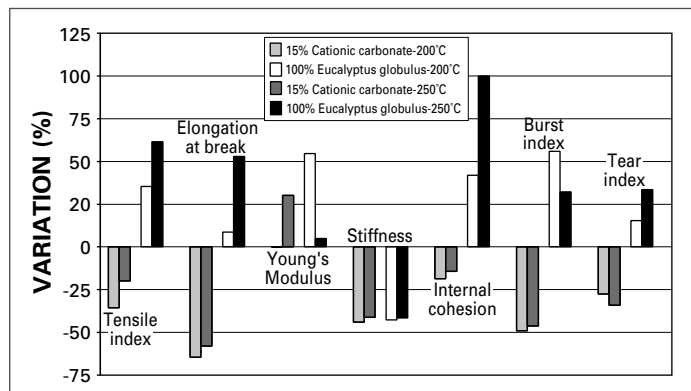
tests confirm the observed delamination for the sheets refined at 25°SR.

Internal cohesion, which detects the presence of some modes of delamination, decreased for higher temperatures, as shown in Fig. 5. The delamination of the samples containing CaCO₃ did not affect optical properties. Although we have signs of delamination, the density of the paper increased, which could explain the decrease in opacity and light scattering coefficient shown in Fig. 6. Although opacity decreased in all paper sets, calcium carbonate can be used to compensate for part of this loss if the sticking and delamination problems can be solved. At all impulse press temperatures, the absolute values of opacity in paper containing CaCO₃ are higher than those observed in paper without additives. The brightness of paper containing CaCO₃ can be improved, which is not the case for the additive-free papers.

CONCLUSIONS

Impulse drying increased the sheet density for papers with cationic starch. Most

IMPULSE DRYING



5. Variation in mechanical properties of impulse-dried samples pressed at 200°C and 250°C with respect to samples pressed at 25°C for additive-free sheets and sheets with 15% CaCO₃ (25°SR).

of the paper strength properties are directly related to sheet density, so higher strengths were also achieved. The presence of cationic starch could be used to compensate for part of the stiffness usually lost in impulse-dried additive-free papers, while maintaining air permeability and smoothness. Some problems of delamination and sticking were observed in the paper sheets with cationic starch. Moreover, paper sheets made from pulps refined to higher degree (40°SR) delaminated more easily than did those made from pulps refined to a lesser extent (25°SR).

Delamination was more pronounced for papers with calcium carbonate than it was for additive-free papers or for papers with cationic starch. Impulse drying negatively affected most of the physical properties of paper containing the mineral filler. If these problems can be solved, introducing calcium carbonate could be a way to compensate for the loss in opacity caused by impulse drying.

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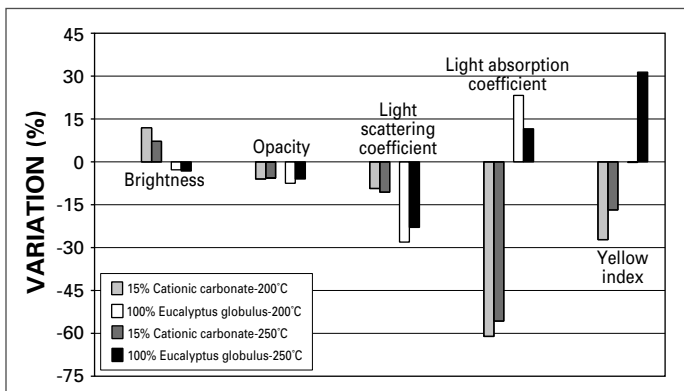
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INSIGHTS FROM THE AUTHORS

Over the past few years, much progress has been made in the field of impulse drying. However, more experimental work should be done to determine how, and if, the combination of additives-containing paper and process can work together to a profitable end.

Although the effect of impulse drying on the properties of free-additive papers are well documented from earlier studies, the effect of this new technology on papers containing different functional additives had not been reported. In fact, this work con-



6. Variation in optical properties of impulse-dried samples pressed at 200°C and 250°C with respect to samples pressed at 25°C for additive-free sheets and sheets with 15% CaCO₃ (25°SR).

stitutes part of a project to investigate the effect of impulse drying on papers containing different wet end additives.

The difficulties we encountered when carrying out this work included keeping the same range of moisture content in different furnishes; cutting the samples while wet, which was solved by placing a plastic frame onto the surface of the forming wire before the introduction of the pulp suspension; finding the right quantities of each additive for each furnish; etc.

Impulse drying has the potential to produce a denser, smoother, and stronger sheet, but these gains are usually accompanied by a detrimental effect on opacity and stiffness. These findings showed that the presence of cationic starch can compensate for part of the stiffness lost, whereas the reduction in opacity can be minimized through the use of calcium carbonate.

With increasing knowledge and understanding of the impulse drying technology, our studies will allow paper mills to apply this emerging technique to improve runnability and realize savings in energy, furnish, and capital.

Further research should focus on solving the problems of paper web sticking and delamination on papers containing cationic starch and calcium carbonate to be able to take the maximum advantage of these additives on impulse dried papers.

—Mohamed Naceur Belgacem

Mendes, Aguilar, Ranita, and Costa are with LEPAE, Dept. of Chemical Engineering, Faculty of Engineering, University of Porto, R. Roberto Frias 4200-465 Porto, Portugal. Belgacem is with École Française de Papeterie et des Industries Graphiques de Grenoble, INPG, BP: 65, 38402 St Martin d'Hères, France. Silvy is with the University of Beira Interior, Dept. of Paper Science and Technology, P-6200 Covilhã, Portugal. Email Belgacem at Naceur.Belgacem@efpg.inpg.fr.



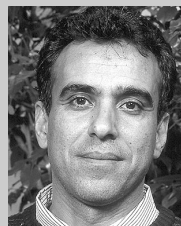
Mendes



Aguilar



Ranita



Belgacem



Silvy



Costa