## EFFECTS OF POLY-ALUMINUM CHLORIDE, STARCH, ALUM, AND ROSIN ON THE ROSIN SIZING, STRENGTH, AND MICROSCOPIC APPEARANCE OF PAPER PREPARED FROM OLD CORRUGATED CONTAINER (OCC) PULP

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The influence of rosin (0.1-0.3%), alum (0.4-0.6%), polyaluminum chloride (0.3-0.7%), and starch (0.5-1.5%) in the making of paper from old corrugated container (OCC) pulp on the freeness, breaking length, tear index, and burst index of pulp and paper sheets was studied. Using a full factorial design to identify the optimum operating conditions, equations relating the dependent variables to the operational variables of the chemical additives were derived that reproduced the former with errors lower than 5%. Using a high starch (1.5%), high PAC (0.7%), low alum (0.4%), and low rosin (0.1%) combination led to pulp that was sufficient to obtain paper with good strength properties (breaking length 5720m; burst index:  $3.1 \text{ kPam}^2\text{g}^{-1}$ ; tear index:  $6.2\text{mNm}^2/\text{g}$ ; Cobb test: 94; fold endurance: 1.52). SEM analysis showed increasing bonding between fibers together at this level of additives. The influence of starch on Cobb test values was not significant.

*Keywords: Old Corrugated Container Pulp; Alum; Rosin; Starch; Papermaking Optimization; Additives; SEM analysis.* 

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### INTRODUCTION

Recycled fiber is an important raw material for the forest products industry, the use of which is growing rapidly. Recycled fiber includes all kinds of recovered papers having natural fiber sources, such as old corrugated container (OCC) pulp, waste newspaper, magazine, and coated board.

Considerable interest has been shown in recent years for the development of drystrength additives having improved efficacy (Lorenćak et al. 2000; Kitaoka and Tanaka 2001; Claesson et al. 2003; Lindström et al. 2005). The present experiments seek a greater understanding – and further increases in performance – of well known drystrength additives. These include of rosin, alum, polyaluminum chloride (PAC), and starch, which is the most widely used wet-end additive for the development of drystrength of papermaking (Fukunaga 1999; Strazdins 1989; Hubbe 2006a, 2006b, 2007).

Despite the fact that these compounds are widely used for enhancing the dry strength of paper and to promote dewatering during the forming process (Ye et al. 1990; Fukunaga 1999), little is known about the optimization of using of these compounds in

terms of the resulting paper from recycled fibers. Furnish containing old corrugated container (OCC) pulp is of particular interest. Therefore, the optimization effects of rosin, aluminum sulfate (papermaker's alum), polyaluminum chloride (PAC), and cationic starch with mathematical models can be interesting to investigate.

Moreover, despite the abundant literature on OCC pulping, no mathematical model appears to have been used to derive equations for predicting the quality of additives as a function of process variables with a view to identifying the most suitable additives conditions.

Most mathematical models applied to the papermaking from raw materials rely on delignification kinetics to predict the extent of delignification (Jiménez et al. 2001, 2002, 2005; Garrote et al. 2003; Wan Rosli et al. 2004; Rezayati Charani et al. 2005, 2006). Such models are complex and impractical when more than two independent variables must be considered. This is avoided by empirical models constructed using an experimental factorial design to estimate different dependent variables for the pulp (e.g. freeness) and strength-related properties of paper sheets obtained from it, as a function of different independent operational variables.

In this work, a full factorial design was used to study the influence of independent variables on the chemical additives (viz. rosin, alum, PAC, and starch percentage) on the freeness, breaking length, burst index, tear index, Cobb test, and fold endurance of the pulp and resulting paper sheets, with the aim of identifying the optimum operating conditions.

### EXPERIMENTAL

#### **Raw Material**

Old corrugated container (OCC) pulp used in the experiments included used cases, sheets, or cuttings of corrugated board, such that the total of unusable materials was no more than 1%. The pH of the furnish was around of 5.

Experiments were conducted by use of industrial water with hardness approx. 155 ppm. Table1 shows the characteristics of additives materials in this research.

Alum	Insoluble Material: 0.2%, Fe <sub>2</sub> O <sub>3:</sub> 36 ppm
Rosin	From Subra rosin company. pH of Solution (2%): 10.5, Specific gravity: 1.08 g/cm <sup>3</sup>
Starch (cationic)	Nitrogen Containing Tapioca Starch Ether(Quaternary); CationicN <sub>2</sub> :0.29- 0.33%; Bulk density:650 gr/l; Moisture content:10-12%; Temperature of dissolution: 73-75 °C; pH of Cold solution (2%): 5.4; pH of Cooked solution (2%): 6.4; Ash of uncooked starch: 0.67%; Viscosity of solution (2%, 23°C): 153ml/g
Poly-aluminum	Al <sub>2</sub> O <sub>3</sub> :28-31%; Basicity:65-85%; Mn<0.0075%;As<0.005; Pb<0.003;
Chionae (FAC)	Hg<0.0002 %

Table 1. Characteristics	of Additives to	OCC Pulp
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### **Characterization of Pulp and Paper Sheets**

The freeness of pulps was measured by TAPPI method T 227 om-94. Handsheets of 120 g/m<sup>2</sup> were formed, and their properties were evaluated in accordance with TAPPI standard methods [TAPPI Committee (2000–2002)]. The handsheets were conditioned at 23 °C and 50% RH for at least 24 h before testing. The Burst Index of handsheets was measured by TAPPI method T 403 om-97. The Breaking length of handsheets was measured by method T 404 cm-92. The Tear index of handsheets was measured by method T 414 om-98. Fold Endurance of handsheets was measured by method T 511 om-96. The Cobb Test of handsheets was measured by method T 402 sp-98.

For Scanning electronic Microscopy (SEM) analysis, handsheets were cut to small samples, mounted on stub with adhesive, and then they were placed under vacuum, evacuated, and sputter-coated with gold. After preparation of samples, the samples were investigated by SEM with a ZEISS DSM 960A (Oberkochen,Germany) instrument.

### Methods

The proposed model used a series of experiments based on a three-level design to estimate the terms of a second-order polynomial equation. The three-level design is written as a  $3^k$  factorial design. This means that k factors are considered, each at 3 levels. These are (usually) referred to as low, intermediate, and high levels. These levels are numerically expressed as -1, 0, and 1. A third level for a continuous factor facilitates investigation of a quadratic relationship between the response and each of the factors. Subsequently, the values of the independent variables were normalized from -1 to +1 by using Eq. (1) in order to facilitate direct comparison of the coefficients and visualization of the effects of the independent variables on the response variable:

$$X_n = 2 \frac{X - \overline{X}}{X_{\text{max}} - X_{\text{min}}} \tag{1}$$

In Eq. (1)  $X_n$  is the normalized value of R, A, P, or S; X is the absolute experimental value of the variable concerned;  $\overline{X}$  is the mean of all the experimental values for the variable in question; and  $X_{max}$  and  $X_{min}$  are the maximum and minimum value, respectively, of such a variable. This normalization also results in more accurate estimates of the regression coefficients as it reduces interrelationships between linear and quadratic terms (Montgomery 1991).

The total number of observations (experiments) required for the three independent variables (viz. Rosin -R-, Alum -A-, PAC -P- and Starch – S-) was calculated from the following equation,

$$N = 3^k \tag{2}$$

and found to be 81. K in the equation is the number of independent variables. The experimental data were fitted to the following second-order polynomial:

$$Z = a_0 + \sum_{i=1}^3 b_i X_{ni} + \sum_{i=1}^3 c_i X_{ni}^2 + \sum_{i=1; j=1}^3 d_{ij} X_{ni} X_{nj} \qquad (i < J)$$
(3)

where Z is the response or dependent variable [viz. Freeness (C.S.F.), Breaking length (BL), Burst index (BI), breaking length (BL), Tear index (TI), Folding Endurance (FE), or Cobb test (CT)];  $X_n$  is the normalized value of the independent variable concerned; and  $a_0$ ,  $b_i$ ,  $c_i$  and  $d_{ij}$  are unknown characteristic constants estimated from the experimental data. Table 2 in the Appendix shows the absolute and normalized values obtained for the independent variables in the 81 tests required to construct the model.

The values of responses obtained allow the calculation of mathematical estimation models for each response, which were subsequently used to characterize the nature of the response surface. All statistical analyses were carried out using the statistical software, MINITAB of Minitab, Inc., USA.

In this way, the MINITAB 15 software suite was used to conduct a multiple linear regression analysis involving all terms in Eq. (3), by use of alpha-to-enter 0.15 and alpha-to-remove 0.15 with the stepwise method. In this method, the p-value was associated with the T-value. The T-value of the predictor equals the coefficient of the predictor divided by the standard error of the coefficient. A larger calculated T-value corresponds to a smaller p-value. The p-value is used to determine whether the predictor is entered or removed from the model. For a predictor outside the model, if the p-value is less than the alpha-to-enter value, and it is the smallest p-value among the predictors outside the model, if the p-value is greater than the alpha-to-enter value, and it is the largest p-value among all the predictors in the model, then the predictor is removed from model.

The independent variables used in the equations relating them to the dependent variables were those having a statistically significant coefficient (viz. those not exceeding a significance level of 0.05 in Student's t-test and having a 95% confidence interval excluding zero). This paper deals with the influence of chemical additives used in the wet end of papermaking machine producing board from old corrugated containers on the freeness and properties of paper sheets such as breaking length, burst index, tear index, fold endurance, and Cobb test in order to determine the best additives conditions.

#### **RESULTS AND DISCUSSION**

All tests were conducted in triplicate. The average results obtained for the dependent variables are shown in Table 3 in the Appendix. Deviations from the means were always less than 10%.

Data processing enabled estimation of the main effects and the interactions of the factors for the responses considered. The effect of a factor is the change in the response when it is changed from the low level (-1) to the high level (+1). The main effect of each factor estimates its average effect over all possible conditions of the other variables. Each of the responses analyzed can be affected only by the main effects or by interactions among them. The main effect of a variable should be individually interpreted only if there is no evidence that the variable interacts with other variables. When there is evidence of one or more such interactions, the interacting variables should be considered jointly.

The equations obtained for the different dependent variables, their R–Squared value (which indicates how well the model fits the data), R-Sq(adj) values, the standard deviation (S), PRESS (the sum of squares of the prediction errors), Mallows' Cp (related to the mean square error of a fitted value), and R-Sq (pred) that indicated how well the model predicts responses for new observations (Minitab Inc 2000-2006), for the terms in such equations (at a confidence level of 95%) were as follows:

$$C SF = 363 .3 + 11 .35 X_{S} + 3.08 X_{P} + 2.55 X_{S}^{2} - 2.71 X_{P}^{2} - 1.82 X_{RP} - 0.61 X_{RA}$$

$$S : 1.37, R - sq : 98 .12, R - sq (adj) : 97 .93, PREES : 140 .944, R - sq (pred) : 97 .62$$
(4)

$$BL = 5512 + 248 .1X_{s} + 61 .8X_{p} + 38 .9X_{A} - 42 .5X_{RA} + 45 .5X_{S}^{2} + 21 .3X_{R} - 21X_{p}^{2} - 12 .4X_{R}^{2} + 10 .3X_{RP} S :15 .7, R - sq : 99 .52, R - sq (adj) : 99 .44, PREES : 20453, R - sq (pred) : 99 .31$$
(5)

 $FE = 1.445 + 0.06X_{s} + 0.029X_{p} - 0.011X_{p}^{2} + 0.007X_{s}^{2} + 0.005X_{A} - 0.0034X_{R}^{2} + 0.0051X_{RP} - 0.004X_{RA}$ (6)

S: 0.0064, R - sq: 98.79, R - sq(adj): 98.62, PREES: 0.00311, R - sq(pred): 98.39

$$TI = 6.338 - 0.107X_s - 0.035X_R - 0.024X_P - 0.022X_R^2 + 0.012X_A^2 - 0.010X_s^2$$
  
S: 0.182, R - sq: 96.74, R - sq(adj): 96.48, PREES: 0.0296, R - sq(pred): 96.07 (7)

$$BI = 2.741 + 0.244X_s + 0.080X_p - 0.061X_R$$

$$S : 0.054, R - sq : 94.32, R - sq(adj) : 94.10, PREES : 0.2503, R - sq(pred) : 93.71$$
(8)

$$CT = 89.24 - 8.295X_{R} - 1.426X_{P} - 2.20X_{R}^{2} - 1.053X_{RP} - 0.179X_{A}$$

$$S : 0.574, R - sq : 99.40, R - sq(adj) : 99.36, PREES : 27.3709, R - sq(pred) : 99.26$$
(9)

where *CSF*, *BL*, *FE*, *TI*, *BI*, and *CT* have the above-described meanings; and  $X_R$ ,  $X_A$ ,  $X_P$ , and  $X_S$  are the normalized rosin, alum, PAC, and starch percentage, respectively.

The values estimated using the previous equations reproduced the experimental values for the different dependent variables with errors less than 3%, 2%, 5%, 1%, 2%, and 2% for freeness, breaking length, fold endurance, tear index, burst index, and Cobb test, respectively.

Identifying the independent variables most (or least) strongly influencing the dependent variables with the previous equations is not so simple as with linear equations, owing to the presence of quadratic terms and interactions between two independent variables. The optimum (greatest) values for the dependent variables, obtained by using the steepest ascent method (Draper and Smith 1981), and the normalized values of the independent variables required to obtain them (in brackets), were as follows: 379ml ( $X_R = -1$ ,  $X_A = 0$ ,  $X_P=1$ ,  $X_S=+1$ ) for the freeness, 5.885km ( $X_R = 0$ ,  $X_A = +1$ ,  $X_P = +1$ ,  $X_S = +1$ ) for the breaking length, 3.126 kPam<sup>2</sup>/g ( $X_R = -1$ ,  $X_A = +1$ ,  $X_P = +1$ ,  $X_S = +1$ ) for the burst index, 6.484 mN m<sup>2</sup>/g ( $X_R = -1$ ,  $X_A = 1$ ,  $X_P = -1$ ,  $X_S = -1$ ) for the tear index, 34 ( $X_R = -1$ ,  $X_A = 0$ ,  $X_P = +1$ ,  $X_S = +1$ ) for the fold endurance and 76 ( $X_R = +1$ ,  $X_A = 0$ ,  $X_P = +1$ ,  $X_S = 0$ ) for the Cobb test.

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Table 4 shows the variations of the dependent variables with changes in each independent variable from -1 to +1 with other variables held constant at the values required to obtain the optimum levels for the dependent variables. Variations are expressed in units of the dependent variables and as percentages (in brackets) relative to their optimum values.

brackets) with changes in the independent variables (non-1 to 1)								
Dependent variables	Independent Variables							
	R	А	Р	S				
Freeness (C.S.F)	3 (0.79%)	1 (0.26%)	9 (2.43%)	22 (6.12%)				
Breaking length (km)	22 (0.37%)	78 (1.34%)	124 (2.14%)	496 (9.21%)				
Tear index (mNm <sup>2</sup> /g)	0.07 (1.08%)	0(0%)	0.32 (0.50%)	0.048 (0.74%)				
Burst index (kPam <sup>2</sup> /g)	0.122 (3.90%)	0 (0%)	0.16(5.12%)	0.49 (15.61%)				
Fold Endurance	0 (0%)	1 (2.13%)	5 (15.49%)	8 (33.44%)				
Cobb Test	19 (19.62%)	0 (0%)	5 (6.02%)	0 (0%)				

**Table 4** Maximum Changes in the Dependent Variables (in units and percentages with respect to the optimum values, which are shown in brackets) with Changes in the Independent Variables (from -1 to +1)

R :Rosin, A : Alum, P :PAC and S: Starch

As can be seen from Table 4, the starch was the independent variable most markedly influencing all dependent variables, except for the Cobb test; the other independent variables had weaker effects. The considerable effect of starch on the dependent variables can be attributed to high amount of defibration and fines in OCC pulp, contributing to a high relative bonded area. It has been stated that dry strength additives increase the strength properties without affecting the sheet structure, but the increase is small when the relative bonded area is small (Retulainen and Nurminen 1996). For instance, Table 4 shows the largest changes in the burst index as an important property of papermaking resulted from variations of the starch percentage (15.61%), which was followed by PAC (5.12%), then rosin (3.90%), while the influence of alum was not significant (0%) under optimum condition. Similar analyses were found for the other dependent variables.

In this research, alum and PAC were used together and the effect of each of them on dependent variables was investigated separately because alum is cheaper than PAC. It is very suitable to use alum instead of PAC, for instance, when improvement in breaking length of paper is needed. However, when it is desired to increase tear index, increasing the alum is not suitable.

Figures were obtained by plotting each dependent variable as a function of the experimental values of each independent variable (all other independent variables change from low to high) in order to show their main effect and interaction of independent variables. Figure 1 shows the main effect of operating conditions on burst index. Similar figures were found for the other dependent variables. It is apparent that the starch was the most positive influential factor in relation to the burst index, followed by the PAC, in order of importance. The effect of alum was comparatively small, but the main influence of rosin had been observably negative. However, the advantage of PAC over alum has

been emphasized in the previous literature (Neimo 1992). These results are confirmed by Eq. 8.



Fig.1. The main effects of the independent variables on burst index

Moreover, Fig. 2 shows the interaction of independent variables on burst index. According to this figure, it can be understood that by variation of starch under every fixed amount of other independent variables, burst index will be changed significantly. Conversely, the interaction between alum under every fixed amount of other independent variables was not as significant as PAC when PAC increases from medium level to high level. There was a considerable positive interaction for PAC when it increased from low level to a medium level, so use of approximately 0.5% PAC seems a sufficient level. The small positive effect of alum here is not identical to what was observed in the previous literature, in which a dry strength decrease due to alum addition was attributed to the formation of aluminum carboxylate linkages between pulp fibers in the dried handsheets that had an additive effect (Saito and Isogai 2005). In the matter of rosin interaction with others as an another factor, it can be concluded from Fig. 2 that there was the same rate of increasing burst index with increasing of rosin under the fixed level of all of other independent variables. These results were confirmed by the burst index equation. Similar figures were found for the other dependent variables.



Interaction Plot (data means) for BI

Fig. 2. The interaction effects of the independent variables on burst index.

On the basis of previous results pertaining to the important effect of starch and PAC on burst index, the regions of interaction between starch and PAC on burst index under fixed amount of other variables are shown separately in Fig 3. According to Fig. 3, it was required to use more than 0.5 % of PAC and 1.45% of starch in order to obtain burst index more than 3kPam<sup>2</sup>/g. Also, by increasing of starch percentage, it was possible to obtain high levels of burst index with the low amount of PAC (0.5%), while in cases where the starch addition was low (<1.20%) it was not possible. It was reported (Kapoor et al. 2001) that the obvious interaction between PAC and starch was the result of higher pH levels (almost > 6.2). It was also observed in the same paper that addition of cationic starch gave improvement in the sizing with dispersed rosin, whereas not much effect was observed for rosin soap size.

A series of extra points (new points) were used to determinate the predictability of equations from 4 to 9 (Table 5). These points included one point as original OCC pulp and some points with fixed percentage of rosin and alum without or with PAC and starch. The values estimated using the previous equations reproduced recent experimental values for the different dependent variables with errors less than 10% for all dependent variables with the exception of 30% for folding endurance. Because of low deviation of these equations, they confirmed that these equations can be assertively applied for prediction of effect of these kinds of additives in papermaking under similar base condition.



Contour Plot of BI vs PAC; STARCH

Fig. 3. The interaction effect of starch and PAC on burst index.

Table 5. Experimental Values of the Dependent Variables in the
Laboratories Result Obtained with the Extra Condition from Experimental
Design Used for OCC Pulp.

		A (0/)	D (0/)	C (0/)	C.S.F(mL	BL	BI(kPam2g-	÷			
Run	R (%)	A (%)	P (%)	5(%)	)	(km)	1)	TI( mNm2g-1	) CT(g/n	n2) FE	
82	0	0	0	0	340	4.833	1.96	6.587	18	1.99	
83	0.2	0.5	0	0	340	4.817	1.92	6.587	18	1.96	
84	0.2	0.5	0.3	0	328	4.823	1.96	6.512	18	1.95	
85	0.2	0.5	0.5	0	323	4.883	1.97	6.489	19	1.95	
86	0.2	0.5	0.7	0	322	4.883	1.97	6.489	19	1.94	
87	0.2	0.5	0	0.5	353	5.117	2.26	6.498	22	1.96	
88	0.2	0.5	0	1	368	5.317	2.45	6.389	24	1.96	
89	0.2	0.5	0	1.5	378	5.567	2.65	6.260	28	1.96	
R, A,	R, A, P and S = Absolute values of rosin, alum, PAC and starch percentage.										

**Table 6.** Values of the Dependent Variables for the Pulp and Resulting Paper

 Sheets (and deviations from the optimum levels) obtained under the conditions

 stated

Dependent variables										
Oper	ation	al co	nditior	IS	C.S.F	BL	BI	TI	СТ	FE
					(mL)	(km)	(kPam <sup>2</sup> g⁻¹)	(mNm <sup>2</sup> g⁻¹)	(g/m <sup>2</sup> )	
<b>a:</b> -1	0	+1	+1	Value of the dependent variable	378	5.807	32	61	93	1.52
				Deviation from the optimum value (%)	0.3	0.07	0.43	0.43	1.74	0.04
<b>b:</b> 0	+1	+1	+1	Value of the dependent variable	378	5.900	31	61	89	1.53
				Deviation from the optimum value (%)	0.2	0.25	0.90	0.90	1.16	0.04
<b>C:</b> -1	-1	-1	-1	Value of the dependent variable	352	5.213	25	64	96	1.34
				Deviation from the optimum value (%)	1.5	0.62	0.62	0.62	0.12	0.07
<b>d:</b> -1	-1	+1	+1	Value of the dependent variable	377	5.720	32	61	94	1.52
				Deviation from the optimum value (%)	0.6	0.49	0.49	0.49	0.86	0.05
<b>e:</b> +1	0	+1	0	Value of the dependent variable	358	5.387	26	62	78	1.41
				Deviation from the optimum value (%)	0.4	0.16	0.69	0.17	0.09	0.01

The following combinations provide the values of the dependent variables (and percent deviations from the optimum levels) shown in Table 6:

- (a) A low-to-medium rosin percentage, medium alum and high PAC, starch.
- (b) A high percentage of alum, PAC, starch and medium rosin.
- (c) Low values of the four additives variables (rosin, alum, PAC and starch).
- (d) A low percentage of rosin, alum, and high PAC and starch.
- (e) A medium percentage of alum, starch and high rosin and PAC.

As can be seen from Table 6, obtaining a pulp with low reduction in freeness entailed using a combination of low alum and high PAC, starch (0.4%, 0.7%, and 1.5%, respectively), and low to medium rosin percentage (0.1% to 0.3%). However, according to the freeness of the original pulp (340 ml) from Table 3, it was confirmed that chemical additives caused changes in the freeness of pulp. The effect of additives, especially cationic starch, on the freeness has been cited in other papers, indicating that starch can remarkably improve dewatering and consequently speed up the drainage (Andersson

1995). So freeness is an important parameter that affects drainage phenomena on a paper machine, especially in the press section, so it needs to be considered when different percentages of chemical additives are applied to modify some paper properties. However, under these conditions, the values of the strength-related properties for the paper sheets departed by only 0.3–2% from their optimum levels; also, in order to increase the burst index of a paper, using of a high percentage of PAC and starch (0.7 % and 1.5%) is essential. It is necessary to note that effect of rosin and alum was negative in this respect. So it is recommended to keep a correct relation between use of rosin and alum versus PAC and starch. In addition, breaking length and folding endurance approximately acted similar to burst index and freeness, while Cobb test results were affected by rosin strongly and then alum, although PAC apparently can be used in place of alum. It was shown that although increased alum dosage might be helpful, the cationic hydrated polyhydroxyaluminum ions and alumina are not efficient enough to create shear-resistant irreversible adsorption of rosin particles (Strazdins 1989). Because of this reason rosin and somehow alum had an almost negative effect on strength properties of paper, or, in other words, they are especially used for controlling the quantity of water absorbed by paper in a specified time under standardized conditions. It is very important to keep a correct relation between use of rosin and alum against to PAC and starch, although the influence of starch on Cobb test results was not significant in terms of a potential saving in chemical cost by adjusting a correct ratio of chemicals. We found the influence of starch on Cobb test results was not significant; however, Wang and Tanaka (2001) reported both aluminum and rosin size content in paper were increased by the addition of suitable polymers. They supposed that suitable cationic polymers can form stable complexes with rosin size particles through hydrogen bonds and with aluminum cations by coordination bonds so as to prevent rosin particles and aluminum cations from the destructive action of OH- ion under neutral-alkaline conditions.

Scanning Electron microscopy (SEM) images are used often for evaluations and analysis of paper structure (Allem 1998; Allem and Uesaka 1999; Dickson 2000; Chinga and Helle 2002; Lipponen et al. 2004). According to obtained results using a combination of high starch (1.5%), high PAC (0.7%), low alum (0.4%), and low rosin (0.1%) led to pulp with good strength properties. A comparison of SEM photos is given in Fig. 4 for OCC pulp without additives, in Fig. 5. for OCC pulp with low starch (0.5%), low PAC (0.3%), high alum (0.6%), and high rosin (0.3%), and in Fig. 6 for OCC pulp with high starch (1.5%), high PAC (0.7%), low alum (0.4%), and low rosin (0.1%), These results prove that increasing in starch level had a large effect on increasing the bonding between microfibers and fibers. Increasing in bonding improves mechanical and strength properties of paper.

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Fig. 4. OCC pulp without additives



Fig. 5. OCC pulp with starch (0.5%), PAC (0.3%), alum (0.6%) and rosin (0.3%)



Fig. 6. OCC pulp with starch (1.5%), PAC (0.7%), alum (0.4%) and rosin (0.1%)

### CONCLUSIONS

1. Using a full factorial design in order to identify the optimum operating chemical additives (rosin, alum, PAC, and cationic starch) for papermaking with old corrugated container (OCC) pulp provided equations that related the freeness, breaking length, burst index, and tear index of the obtained paper sheets, with the operational variables, and predicted the former with errors less than 5% for all dependent variables.

2. Using a combination of high starch (1.5%), high PAC (0.7%), low alum (0.4%), and low rosin (0.1%) led to pulp conditions that yielded paper with good strength properties, but provided paper sheets that will have relatively high Cobb test values, which may restrict their applications. The combination of rosin and alum tended to negatively affect strength properties of paper; in the other words, these additives mainly are used for controlling the quantity of water absorbed by paper in a specified time under standardized conditions. It is very important to keep a correct relation between use of rosin and alum versus PAC and starch. Therefore, there is a potential saving in chemical cost by adjusting the ratio of chemicals.

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### APPENDIX

**Table 2.** Absolute and Normalized Values of the Operational Variables Used in the Laboratories Result Obtained with the Experimental Design Used

Experiment	<i>R</i> (%)	A(%)	P (%)	S (%)	X <sub>R</sub>	X <sub>A</sub>	$X_{P}$	Xs
1	0.1	0.4	0.3	0.5	-1	-1	-1	-1
2	0.1	0.4	0.3	1	-1	-1	-1	0
3	0.1	0.4	0.3	1.5	-1	-1	-1	1
4	0.1	0.4	0.5	0.5	-1	-1	0	-1
5	0.1	0.4	0.5	1	-1	-1	0	0
6	0.1	0.4	0.5	1.5	-1	-1	0	1
7	0.1	0.4	0.7	0.5	-1	-1	1	-1
8	0.1	0.4	0.7	1	-1	-1	1	0
9	0.1	0.4	0.7	1.5	-1	-1	1	1
10	0.1	0.5	0.3	0.5	-1	0	-1	-1
11	0.1	0.5	0.3	1	-1	0	-1	0
12	0.1	0.5	0.3	1.5	-1	0	-1	1
13	0.1	0.5	0.5	0.5	-1	0	0	-1
14	0.1	0.5	0.5	1	-1	0	0	0
15	0.1	0.5	0.5	1.5	-1	0	0	1
16	0.1	0.5	0.7	0.5	-1	0	1	-1
17	0.1	0.5	0.7	1	-1	0	1	0
18	0.1	0.5	0.7	1.5	-1	0	1	1
19	0.1	0.6	0.3	0.5	-1	1	-1	-1
20	0.1	0.6	0.3	1	-1	1	-1	0
21	0.1	0.6	0.3	1.5	-1	1	-1	1
22	0.1	0.6	0.5	0.5	-1	1	0	-1
23	0.1	0.6	0.5	1	-1	1	0	0
24	0.1	0.6	0.5	1.5	-1	1	0	1
25	0.1	0.6	0.7	0.5	-1	1	1	-1
26	0.1	0.6	0.7	1	-1	1	1	0
27	0.1	0.6	0.7	1.5	-1	1	1	1
28	0.2	0.4	0.3	0.5	0	-1	-1	-1
29	0.2	0.4	0.3	1	0	-1	-1	0
30	0.2	0.4	0.3	1.5	0	-1	-1	1
31	0.2	0.4	0.5	0.5	0	-1	0	-1
32	0.2	0.4	0.5	1	0	-1	0	0
33	0.2	0.4	0.5	1.5	0	-1	0	1
34	0.2	0.4	0.7	0.5	0	-1	1	-1
35	0.2	0.4	0.7	1	0	-1	1	0
36	0.2	0.4	0.7	1.5	0	-1	1	1
37	0.2	0.5	0.3	0.5	0	0	-1	-1
38	0.2	0.5	0.3	1	0	0	-1	0
39	0.2	0.5	0.3	1.5	0	0	-1	1
40	0.2	0.5	0.5	0.5	0	0	0	-1
41	0.2	0.5	0.5	1	0	0	0	0
42	0.2	0.5	0.5	1.5	0	0	0	1
43	0.2	0.5	0.7	0.5	0	0	1	-1
44	0.2	0.5	0.7	1	0	0	1	0
45	0.2	0.5	0.7	1.5	0	0	1	1

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Experiment	<i>R</i> (%)	A(%)	P (%)	S (%)	X <sub>R</sub>	X <sub>A</sub>	X <sub>P</sub>	Xs
46	0.2	0.6	0.3	0.5	0	1	-1	-1
47	0.2	0.6	0.3	1	0	1	-1	0
48	0.2	0.6	0.3	1.5	0	1	-1	1
49	0.2	0.6	0.5	0.5	0	1	0	-1
50	0.2	0.6	0.5	1	0	1	0	0
51	0.2	0.6	0.5	1.5	0	1	0	1
52	0.2	0.6	0.7	0.5	0	1	1	-1
53	0.2	0.6	0.7	1	0	1	1	0
54	0.2	0.6	0.7	1.5	0	1	1	1
55	0.3	0.4	0.3	0.5	1	-1	-1	-1
56	0.3	0.4	0.3	1	1	-1	-1	0
57	0.3	0.4	0.3	1.5	1	-1	-1	1
58	0.3	0.4	0.5	0.5	1	-1	0	-1
59	0.3	0.4	0.5	1	1	-1	0	0
60	0.3	0.4	0.5	1.5	1	-1	0	1
61	0.3	0.4	0.7	0.5	1	-1	1	-1
62	0.3	0.4	0.7	1	1	-1	1	0
63	0.3	0.4	0.7	1.5	1	-1	1	1
64	0.3	0.5	0.3	0.5	1	0	-1	-1
65	0.3	0.5	0.3	1	1	0	-1	0
66	0.3	0.5	0.3	1.5	1	0	-1	1
67	0.3	0.5	0.5	0.5	1	0	0	-1
68	0.3	0.5	0.5	1	1	0	0	0
69	0.3	0.5	0.5	1.5	1	0	0	1
70	0.3	0.5	0.7	0.5	1	0	1	-1
71	0.3	0.5	0.7	1	1	0	1	0
72	0.3	0.5	0.7	1.5	1	0	1	1
73	0.3	0.6	0.3	0.5	1	1	-1	-1
74	0.3	0.6	0.3	1	1	1	-1	0
75	0.3	0.6	0.3	1.5	1	1	-1	1
76	0.3	0.6	0.5	0.5	1	1	0	-1
77	0.3	0.6	0.5	1	1	1	0	0
78	0.3	0.6	0.5	1.5	1	1	0	1
79	0.3	0.6	0.7	0.5	1	1	1	-1
80	0.3	0.6	0.7	1	1	1	1	0
81	0.3	0.6	0.7	1.5	1	1	1	1

*R*, *A*, *P*, and *S* = Absolute values of rosin, alum, PAC and starch percentage  $X_{\rm R}$ ,  $X_{\rm A}$ ,  $X_{\rm P}$ , and  $X_{\rm S}$  = Normalized values of rosin, alum, PAC and starch percentage

**Table 3**. Experimental Values of the Dependent Variables in the Laboratory

 Results Obtained with the Experimental Design Used.

Exp.	C.S.F	BL	BI (kPam <sup>2</sup> g <sup>-1</sup> )	TI 2 -1	CT	FE
	(mL)	(km)	( a g )	( mNm <sup>2</sup> g <sup>-1</sup> )	(g/m²)	
1	352	5.213	2.480	6.493	96	1.34
2	355	5.390	2.758	6.378	96	1.36
3	368	5.690	2.911	6.289	96	1.45
4	353	5.307	2.637	6.459	96	1.38
5	362	5.417	2.826	6.344	96	1.43
6	378	5.683	3.114	6.255	96	1.51
7	357	5.247	2.614	6.452	95	1.38
8	367	5.480	2.794	6.350	95	1.45
9	377	5.720	3.127	6.194	94	1.52
10	355	5.230	2.457	6.483	95	1.36
11	357	5.397	2.709	6.378	96	1.40
12	372	5.697	2.895	6.245	96	1.46
13	353	5.257	2.627	6.452	96	1.38
14	362	5.477	2.816	6.340	96	1.45
15	378	5.743	3.124	6.238	95	1.52
16	357	5.293	2.591	6.442	95	1.41
17	363	5.513	2.810	6.344	94	1.46
18	378	5.807	3.117	6.218	93	1.52
19	357	5.347	2.476	6.456	96	1.36
20	355	5.463	2.767	6.357	96	1.40
21	368	5.763	2.934	6.303	96	1.52
22	353	5.370	2.630	6.476	95	1.40
23	362	5.553	2.823	6.337	96	1.46
24	378	5.773	3.183	6.248	95	1.53
25	358	5.417	2.601	6.435	94	1.43
26	362	5.593	2.823	6.364	94	1.46
27	368	5.877	3.144	6.187	94	1.52
28	348	5.217	2.349	6.463	90	1.36
29	358	5.383	2.650	6.429	90	1.40
30	372	5.683	2.833	6.296	90	1.46
31	355	5.250	2.552	6.456	90	1.38
32	362	5.467	2.748	6.347	90	1.45
33	373	5.750	3.042	6.245	89	1.51
34	357	5.283	2.552	6.442	88	1.40
35	363	5,493	2.748	6.327	89	1.45
36	377	5.797	3.039	6.207	89	1.52
37	350	5.200	2.355	6.442	90	1.36
38	358	5.450	2.650	6.364	91	1.40
39	370	5 733	2 846	6 224	91	1 48
40	353	5 300	2 552	6 439	90	1 40
41	360	5 533	2 748	6 327	90	1 45
42	378	5 833	3 042	6 224	90	1.10
43	355	5.333	2.565	6.429	88	1 41
44	360	5 550	2 761	6 327	89	1 46
45	378	5 850	3 075	6 156	89	1.53
46	352	5 267	2 362	6 4 5 9	90	1.38
40	362	5 500	2.656	6.367	90	1.00
	002	0.000	2.000	0.007	50	1.71

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Exp.	C.S.F	BL	BI (kPam <sup>2</sup> g <sup>-1</sup> )	<b>TI</b>	CT	FE
	(mL)	(km)		( mNm <sup>•</sup> g <sup>•</sup> )	(g/m²)	
48	372	5.783	2.846	6.241	90	1.48
49	355	5.350	2.561	6.442	89	1.40
50	363	5.560	2.754	6.337	89	1.45
51	378	5.843	3.042	6.231	89	1.53
52	357	5.383	2.571	6.429	88	1.41
53	363	5.600	2.764	6.327	88	1.46
54	378	5.900	3.078	6.173	89	1.53
55	350	5.177	2.303	6.398	82	1.34
56	360	5.437	2.633	6.299	82	1.40
57	368	5.727	2.816	6.187	82	1.46
58	357	5.333	2.483	6.378	78	1.38
59	367	5.530	2.679	6.313	78	1.43
60	378	5.827	2.987	6.190	78	1.52
61	358	5.387	2.519	6.344	78	1.41
62	362	5.553	2.702	6.265	77	1.46
63	375	5.863	2.983	6.163	76	1.53
64	351	5.177	2.290	6.408	82	1.34
65	362	5.420	2.630	6.327	82	1.40
66	372	5.717	2.761	6.187	82	1.45
67	353	5.320	2.493	6.374	78	1.38
68	362	5.530	2.659	6.276	78	1.43
69	378	5.810	2.977	6.150	78	1.51
70	362	5.390	2.499	6.330	77	1.41
71	363	5.563	2.695	6.245	76	1.46
72	375	5.760	2.996	6.173	76	1.53
73	348	5.183	2.323	6.422	82	1.34
74	363	5.417	2.627	6.327	82	1.40
75	372	5.727	2.816	6.190	82	1.46
76	352	5.303	2.627	6.398	78	1.38
77	363	5.527	2.692	6.296	78	1.45
78	378	5.797	3.016	6.194	78	1.52
79	358	5.370	2.529	6.354	76	1.41
80	362	5.533	2.722	6.262	76	1.46
81	373	5.867	3.016	6.163	75	1.52

Exp.: Experimental, CSF: Freeness, BL: Breaking length, BI: Burst index, TI: Tear index,

FE: Fold Endurance, CT: Cobb test