Statistical Evaluation of the Effect of Refining on Fibre Charge of Bagasse Pulp

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ABSTRACT

The aim of this work was to study the influence of refining on various properties of bleached bagasse pulp such as freeness, specific surface area, specific volume, surface charge, total charge and paper properties. These parameters are quantitatively determined for pulp freeness levels between 225-430 ml CSF, which are important for approach flow, and wet end paper making operations. The results on refining conducted in PFI mill indicated that specific surface area and specific volume of the pulps as determined by permeability tester and water retention value (WRV) as determined by Centrifuge method increased with increased refining. The surface charge, as determined by particle charge detector using poly-DADMAC (poly diallyl dimethyl ammonium chloride), also increased with refining. However, the total fibre charge, as determined by conductometric titration, is not affected by refining. The increases in specific surface area of pulps by refining resulted in a higher fibre surface charge and also better fibrefibre bonding. The experimental data are subjected to statistical regression analysis to develop linear univariate regression models, which are found to be accurate with regression coefficients, R^2, close to unity. The comparison of model predicted data and the experimental data shows an excellent agreement between them.

Key words: Beating/refining, Surface Charge, Total Charge, Specific Surface Area, Specific Volume, Water Retention Value (WRV) and Paper Properties.

LITERATURE REVIEW

The cellulosic fibres normally carry a negative charge when suspended in water due to the presence of ionisable acidic groups in the hemicelluloses and lignin. The charge of fibres is a complex function of the chemical composition, state of ionization of the acid groups, and the nature and amount of additional substances adsorbed on the fibre surface. The population of ionisable groups depends on the origin of the fibres and on the chemical treatments such as pulping and bleaching. The characteristics of any particular fibre surface also depend greatly on the degree of mechanical treatment. Cellulosic fibres for papermaking are normally subjected to a mechanical treatment usually known as beating or refining. The effects of refining are most often evaluated by the freeness tests. The four main actions of refining are: internal fibrillation caused by the breakdown of fibre walls into separate lamellas which increases the flexibility of fibres; external fibrillation described as the creation and/or exposure of fibrils on the surface of the fibres; fibre cutting; and abrasion of the surface at the molecular level to produce a more gelatinous surface. The specific surface area and specific volume have been used to measure the primary effects caused by refining of pulps. If increased surface area due to mechanical action leads to more adsorption capacity in direct proportion to the increase in surface area, then as beating opens up the fibre surface, the fibre surface charge should increase with increased beating. This fibre surface charge is also shown to greatly influence the fibre swelling and paper strength properties [1-2]. Refined fibres have higher water retention and stronger inter-fibre bond strength [3].

The properties of paper are highly dependent on the properties of the pulp fibres. In addition to the specific surface area, the bonding ability of single fibres is highly relevant to the physical strength of the paper produced. The fibrefibre bonding strength is affected by the electrokinetic properties of the fibres, especially those related to surface charge [4]. The fibre hydrophilicity and swelling, which are highly relevant to fibrefibre bonding, are also affected by fibre charge [2].

The refining of any fibrous raw material is a complex process and the most influential of all of the papermaking processes. Mechanical treatment of chemical pulps has been reviewed [6]. Many structural changes to the fibres are attributed to beating, increased fibre swelling, fibre shortening, internal & external fibrillation, etc. [7]. Apart from improving fibre properties, it has the greatest influence on product quality. Refining of pulp significantly contributes to papermaking process by affecting not only the quality of end product but also the runnability of the stock. A correct approach towards refining treatment is very essential for product with desired properties.

In papermaking, beating to improve bonding and develop optimum strength properties deliberately enlarges the fibre surfaces. As fibre surfaces are peeled off during beating/refining, increasing amounts of charged fines are also formed. The primary fines consisting of parenchyma cells tend to contribute bulk and some opacity to a sheet of paper whereas the secondary fines produced by delaminating the outer layers of fibre during beating/refining tend to be slender and flexible ideal for bonding. It is evident that the effect of beating on pulp charge is more complex than the effect of chemical composition of pulp as both chemical and mechanical factors are involved. More studies are needed to understand the effect of beating on pulp charge. The individual contribution of fibres & fines to charge of pulp needs to be assessed and the better control of surface charge during beating of pulp

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may result in improved fibre-fibre bonding. So, better understanding of the mechanism of charge development is desired.

A few studies have been carried out [5] earlier on the influence of refining on freeness, specific surface area, specific volume, surface charge, total charge, water retention value and Hand sheet preparation, and compare these results obtained from experiments with different beating of pulps. Attempts have been made to establish correlations between the above mentioned pulp properties for bagasse pulp.

EXPERIMENTAL

Chemicals:

The analytical grades of NaOH, HCl and NaCl were used in the experiments. Polydiallyldimethyl ammonium chloride (Poly-DADMAC, molecular weight 150200,000 g/mol) and sodium-polyethylenesulphate (PES-Na) were reagent grades. These chemicals were diluted with deionised water with a conductivity of 8×10^{-4} mS/cm to the desired concentration before use.

Sources and preparation of pulps:

The dryness of the bleached bagasse pulp received from Central Pulp and Paper Research Institute (CPPRI) was 25%. The bleached bagasse pulp was beaten in a PFI Mill after dilution with distilled and deionised water to different freeness levels following Tappi test method T 200 sp-96. The beaten pulps were filtered on a Buchner funnel using a laboratory vacuum pump to form a wet fibre pad and the filtrate recirculated once to avoid loss of fines. Pulp freeness was measured using standard Tappi test method T 227 om-99 for the determination of Canadian Standard Freeness.

Before the charge measurements were undertaken, the pulp samples were converted to their fully protonated form by soaking the redispersed pulp pad at 1% consistency in 0.01M hydrochloric acid for 16 h as suggested by a previous researcher [8]. The pulp pH after 16 h of soaking was close to 2.2. The pulp was then vacuum filtered using a Buchner funnel and washed several times with deionised water until the pH of the water filtrate was close to 6.0. The vacuum was maintained until no more water could be extracted from the pulp mat. After each titration, the amount of fibre in each sample was determined gravimetrically by filtering the pulp on pre-weighed filter paper and drying in an oven at 105 ^{0}C until a constant weight was obtained.

Determination of charge

Conductometric titrations:

The conductometric titration has been outlined by Katz et al. [9] and is based on changes in conductance of the suspension, which in turn is related to the concentration of the most highly conducting ions, i.e. the hydrogen and hydroxyl ions. Approximately 3 g O.D. pulp converted to hydrogen form is dispersed in 450 ml of 0.001 M sodium chloride prepared in deionised water and addition of 0.5 ml of 0.05M HCl was made before the start of titration. Conductometric titration is carried out with 0.1 M sodium hydroxide dispensed from a micro-burette while the suspension is stirred. Conductivity measurements were made every 1 min after 0.1 ml of alkali solution addition except near the point of inflection where readings were taken after every 0.05 ml of alkali. The conductivity decreased until excess acid was neutralized. Further additions of alkali reacted with the acid groups bound to the pulp and did not change the conductivity. Excess alkali again raised the conductivity. The titration gave the conductivity of the suspension as a function of the volume of alkali added, and the additional volume of alkali needed to reach the second inflection point after the first inflection point gave the carboxylic acid content.

Polyelectrolyte titrations:

The polyelectrolyte titrations were performed using a MUTEK particle charge detector (PCD-02).

The pulp sample (0.5 g) was diluted with 100 g of 0.001N Poly-DADMAC and stirred by a magnetic stirrer for 2 h. During this time the cationic polyelectrolyte completely neutralized the anionic charge in the pulp. The solid content of the pulp was removed on a nylon sieve and 10 ml of the filtrate was pipetted into the cell of PCD-02 (MUTEK) and titrated with 0.001N PES-Na to the endpoint where the streaming potential reached 0 mV. The rate of titration was controlled at 0.3 ml/min. In addition 10 ml of Poly-DADMAC solution was titrated with PES-Na to the neutral point to determine the blank value. In this investigation all charge determinations were made at a 0.001M NaCl concentration. The specific charge density of the sample was calculated by the formula below:

$$q = \frac{(V_p - V_b)xcx100}{w}$$

Where q is the specific charge density (mmol/kg), V_p is the volume of titrant used for pulp (ml), V_b is the volume of titrant used for blank (ml), c is the concentration of titrant (mol/l), and w is the solid content of pulp (g).

Measurement of Specific surface area and specific volume:

The specific surface area and specific volume of the pulp were measured according to the Pulmac manual instructions. Approximately 6 g OD pulp was diluted with deionized water to 0.5% consistency for each test. For the measurement, a uniform pulp mat of about 20 mm thickness with gentle compression was formed from dilute and deaerated slurry by filtration at a carefully controlled rate. A permeable piston was lowered until it rested on the top face of the pulp mat before the compression stage. With a spacer, the pulp mat could be compressed to a desired thickness. Subsequently, the water permeation of the compressed pulp mat was measured using an upward flow through the pulp mat. The flow rate was established and measured by means of a rotameter. The pressure drop across the bed was measured using a manometer. In each test the pressure drop and flow rate for at least six different degrees of compression was recorded. Following the test, the pulp pad was dried in an oven at 105^{o}C until a constant weight was obtained. These data were then used to calculate the equivalent specific surface area and the specific volume of the pulp using the following equation (Robertson and Mason, 1949):

$$\left[\frac{C}{R_p}\right]^{1/2} = \left[\frac{1}{ks^2_w}\right]^{1/2}(1 - VC)$$

$$\text{With } R_p = \frac{A^2\Delta P}{q(w)\eta} \text{ and } c = \frac{W}{AL}$$

Where R_p -Specific resistance to permeation, cm/gm; A - Pad area,

cm^2; ΔP- Pressure drop, dynes/ cm^2; q- Flow rate, cm^3/sec; W- Pad weight, gm; η- Viscosity, poises (gm./cm/sec.); L- Pad thickness, cm; K- Kozeny constant; S_w - Pulp specific surface, cm^2/gm; v- Pulp specific volume, cm^3/gm; c- Pad density, gm/cm^3.

Water retention value (WRV):

The WRV of the pulp is measured through centrifuge method. The pulp sample 0.18 g O.D. basis is slurried with 18 ml water. The slurry is divided into four small cylinders having 60 mm mesh and weight of 38.785 gm. The four cylinders are gently placed in bowl of centrifuge. The operation starts and continues for 30 min. at 2400 No. Thereafter the pulp is weighed. The pulp then left for oven drying and again oven-dried weight is measured. Using the fallowing formula the WRV of the pulp is measured.

$$WRV = \frac{\text{wet weight- Dry weight}}{\text{wet weight}} x100$$

Lab hand-sheet preparation:

Bleached pulp (300 g O.D) beaten in a PFI beater to a different freeness levels and the pulp is diluted in 2000 ml fresh water and then disintegrated at 3000 No. for about 3 minutes. Pulp slurry equivalent to 1.2 g O.D solid is taken in British sheet former for the preparation of approximately 60 g/m2 paper hand sheet. The hand sheets are pressed in hydraulic press at 20 kg/cm2 pressures and then air- dried. After making sheets, they were allowed to dry in open atmosphere minimum for 24 hours. Finally hand sheets are tested for all properties as per standard. Tappi testing method T 205 om-88 carries out the process.

RESULTS AND DISCUSSION

Each refined pulp sample was analysed to obtain freeness, surface charge, specific surface area and specific volume. The conductometric titration was used to measure the total charge of pulp whereas the polyelectrolyte titration was used to measure the surface charge of pulp as accessible to poly-DADMAC. The average fibre charge values of pulps were obtained from at least three tests on each sample. The centrifuge method was used to measure water retention value of the pulp. The strength properties (tensile index, breaking length, tear index and burst index values) of the handmade sheet made from British sheet former are compared as a function of CSF with revolutions as parameter. The Characterization of the parameters analysed for the various refined pulps are shown in Table-1. From the plethora of data, various figures are also drawn to show the parametric effect on the properties of paper with surface and total charge as the major parameters. These are discussed in the following paragraphs.

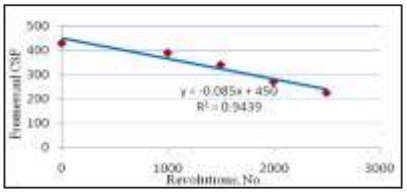
Relationship between freeness and revolutions:

The values of CSF are shown in **Table-1**. In **fig. 1** pulp freeness is represented as a function of revolutions. It is apparent that the pulp freeness decreased with various revolutions. **Fig.1** depicts freeness vs revolutions, which clearly show that the relationship is linear but negatively (decreasing) trend.

Table-1: Characterization of various refined pulps

Revolutions, No.	Freeness, ml CSF	Surface charge, µeq/g	Total charge, µeq/g	Charge ratio,%	Sp. Surface Area,m^2/g	Sp. Volume, cm^3/g	WRV, %	Tensile index, N.m/g	Tear index, mN.m^2/g	Burst index, kPa.m^2/g
0	430	9.95	92.81	10.7	2.09	3.21	176	45.93	4.74	2.30
1000	390	13.65	93.98	14.5	2.69	3.50	204	49.30	5.39	3.14
1500	340	17.04	91.12	18.7	2.84	3.84	232	52.59	5.06	3.60
2000	270	24.14	90.90	26.5	3.44	4.69	256	55.13	4.74	3.75
2500	225	29.87	92.10	32.4	3.50	4.75	278	58.88	4.65	4.29

Fig. 1: The relation between Freeness and Revolutions of various refined pulps.



The effect of refining on fibre surface charge and total charge of pulp:

The values of surface charge of refined pulps are shown in **Table-1** is plotted in **Fig. 2.**The values are compared at a pulp freeness of about 225- 430 ml CSF. The unbeaten pulp was the lowest surface charge about 9.95µeq/g. At 1500 No. Beating pulp had the moderate surface charge and total charge, 17.042 and 91.12µeq/g, respectively and at 2500 No. Beating pulp had highest surface charge and the total charge, 29.87 and 92.1µeq/g, respectively. However, the Charge ratio (surface/total) is the lowest, 0.107, for unbeaten pulp and the highest, 0.321, for 2500 No. Beating pulp. The charge ratio is a function of freeness is shown in **fig. 3.** For the pulps of different freeness, the fibre surface charge increased linearly with increased refining level. As refining opens up the fibre surface, the fibre surface charge increases. Refining does apparently not change the total charge of beating. This observation suggested that the total charge depended mainly on the chemical composition of the fibre wall but not on the physical change of the fibre. In figure 3 shows, the charge ratio increases with increasing the surface charge and regression coefficient is 0.99.

The papermaking fibres are porous and polymers diffuse into them, lower molecular weights penetrating faster and to a greater extent [10]. The results of fibre charge measurement utilising different molecular weight poly-DADMAC showed that the low molecular weight (8750 Mw) polymer could reach all the charged sites in fibre wall whereas medium (48,000 Mw) and high molecular weight (1,200,000 Mw) polymers could only reach the charge sites available onto certain depth of fibre wall [3]. When poly-DADMAC was used, the (surface charge/total charge) ratios are within 0.107 0.321 depending on the fibre type and the molecular weight of the polymer used.

Fig. 2:
Effect of refining on Surface charge of pulps of various Freeness level.

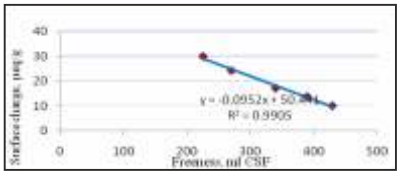
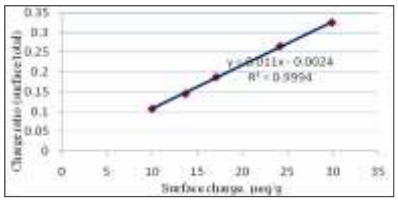


Fig. 3:
The relation between Charge ratio and Surface charge of various refined pulps.



The effect of refining on specific surface area and specific volume:

The specific surface area and specific volume are the pulp properties that influence the characteristics of paper product. The specific surface area characterizes the amount of surface area per unit weight that a wet pulp exhibits whereas the specific volume characterizes the swollen volume of the pulp on a unit weight basis. **Table-1** depicts average surface area, and specific volume as a function of freeness. For all the beating pulps analysed, the specific surface area of pulps increased linearly with further refining treatment (decreased pulp freeness) of the same pulp, consistent to the fact that refining of pulp induced fibrillation of fibres and formation of fines. Both contributed to the increase of specific surface area. In **fig. 4** and **fig. 5**, the specific surface area and specific volume is increased with decreasing the various freeness levels. The results obtained from experiments with the different beating pulps revealed that the specific volume of pulp increased with increasing refining for all the beating pulps. The specific volume of pulp increased from 3.21 to 4.75cm3/g for different beating pulps. It should be noted that severe refining could lead to fibre shortening; resulting in increased specific surface area of the pulps, and subsequently increased the fibre surface charge. For all the pulps, the surface charge is found to be approximately proportional to the specific surface area.

Fig. 4:
Effect of refining on Specific surface area of pulp of various freeness level.

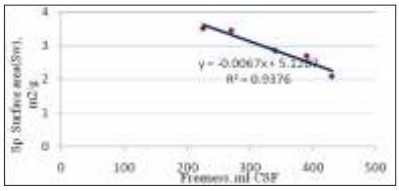
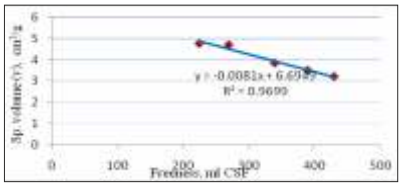


Fig. 5:
Effect of refining on Specific volume of pulp of various freeness level.



The effect of refining on water retention value (WRV):

The water retention value at various freeness levels is shown in **Table-1.** Initially the unbeaten pulp of bleached baggase pulp had low CSF value at about 156.66%. After increasing the revolutions, the water retention values are increased up to 278.40%. The difference between water retention values of unbeaten samples and beating samples is high which shows that the fibrillation of fibres is more in this case. The effect of refining on water retention values at different freeness is shown in **fig. 6.**

WRV is increased with increasing the revolutions and also decreasing the freeness levels. WRV data is generally used in the measurement of water content as a percentage of oven dry weight. Results describe the higher initial beaten fibres has the higher water retention value. As fibres are beaten the cell walls are delaminated and the microspores are created. Result could indicate that the portion of internally laminated fibre cell wall exist much more in higher beaten fibres as water retention value is consistent increases with beating as shown in **Fig.6.** Also beating increases the pulp drainage resistance increased due to an increased in surface area (mainly fines formation). Internal fibrillation increases the fibre swelling and flexibility by loosening the cell wall structure and external fibrillation increases the outer surface of fibres. WRV is depending on the surface charge. According to surface charge the WRV are increased is shown in **fig. 7.**

Fig. 6:
Effect of refining on Water retention value (WRV) of pulp of various freeness level.

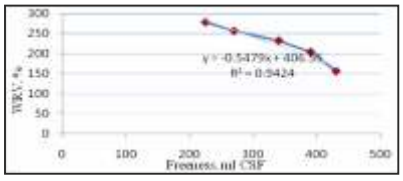
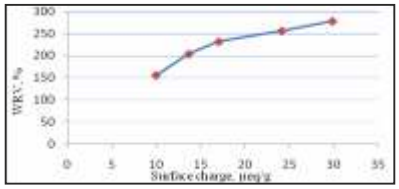


Fig. 7:
Water retention value vs Surface charge of various refined pulps.



The effect of refining on paper properties:

In Table-1 the strength properties (tensile index, breaking length, tear index and burst index values) of the handmade sheet made from British sheet former are compared as a function of CSF with revolutions as parameter.

The effect of refining on tensile index:

The Table-1 shows characterization of different parameters at various freeness levels. In **figs 8** and **9** shows the effect of refining on tensile index of beating pulps and surface charge. According to different beating pulps the tensile index is increases and at freeness levels the tensile index is decreased. In **figure 9** shows, tensile index increases with increasing the surface charge.

Fig. 8:
Effect of refining on tensile index of pulp of various freeness level.

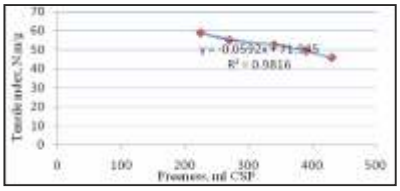
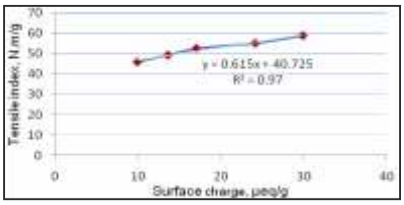


Fig. 9:
Tensile index vs Surface charge of different refined pulps.



The effect of refining on tear index:

The Table-1 shows characterization of different parameters at various freeness levels and revolutions. In case of tear index initially for breaking of fibre-to-fibre bonds the tear strength requirement is high. As the beating increases the fibre fibre bonds get broke which result in requirement of less tear strength. According to the relationship between tensile index and various freeness levels is similar to the relationship between tear index and various freeness levels. Whereas tear index was slightly increased and then decrease with freeness levels. Similarly the relationship between tear index and surface charge, the tear index

is slightly decreased with compared to surface charge.

The effect of refining on burst index:

The Table-1 shows characterization of different parameters at various freeness levels and revolutions. According to the relationship between tensile index and various freeness levels is similar to the relationship between burst index and various freeness levels. Whereas burst index was decreased and then decrease with freeness levels. According to different beating pulps the burst index is increases and at freeness levels the burst index is decreased. Similarly the relationship between burst index and surface charge, the burst index is increased with compared to fibre surface charge. The pulps which are high in hemicelluloses beat more rapidly than those low in hemicelluloses. High hemi cellulosic content produces paper that is stronger in burst and tensile strength. This is consistent with the concept that the highly hydrophilic hemicelluloses absorb water, thereby causing fibre to swell and become flexible; the swelling results in weakening of bonds between fibrils, facilating fibrillation under mechanical treatment [11]. The retaining of baggase at 30 meshes in nearly same amount and low water retention value leads to the high tensile and burst index.

Statistical Interpretation of Data

Based on the experimental data obtained from the laboratory are analysed from graphs and then subjected to statistical regression analysis. Least square techniques have been used. For simplicity linear regressions have been tried. Some important univariate linear regressions equations are developed. However, some are found to be distinctly linear whereas some possesses slight nonlinear characteristics. Predicted data from regression models are plotted against experimental values in various graphs. Percentage error is defined as [(the experimental value model predicted value)/ experimental value] * 100. These regression models along with the maximum and minimum percentage error are depicted in **Table 2.**

It is evident that all the regression models developed are very accurate as the regression coefficients, R^2 are close to unity (on an average above 0.98) and the percentage error does not exceed in any case ± 7%. The model predicted data and the experimental data are compared in various graphs. A few of them are shown as typical examples. The plots between models predicted data and experimental data show an excellent agreement between them. Therefore statistical predictions are claimed to be very good and reproducible with a reasonable degree of accuracy permitted in engineering estimates. Therefore the statistical regression can be used reliably for analysis purposes.

The model predicted data and the experimental data are compared in graphs are as follows to example:

Example: Surface Charge Vs Freeness

Regression equation and regression coefficient are

$y = -0.095x + 50.44, R^2 = 0.9905$

$$\% \text{ Error} = \frac{\text{Experimental value-Model predicated value}}{\text{Experimental value}} \times 100$$

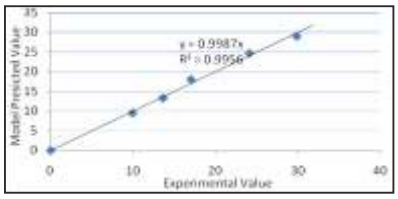
The model predicted data and the experimental data are shown in **table 3**.

Fig. 10 Depicts the relationships between the experimental value and model predicted values at

$y = -0.095x + 50.44, R^2 = 0.9905$.

Similarly, the experimental value and model predicted values at different

Fig. 10:
The experimental value and model predicted values at
y = -0.095x + 50.44.



Regression equations and regression coefficients are similar to **fig. 10.**

CONCLUSION

Refining operation increases surface charge, specific surface area and specific volume of fibres, but did not change the total fibre charge or in other words, the total fibre charge is not affected by refining, but the fibre surface charge increases with the degree of refining. The increase in specific surface area as well as the increase in surface charge of the fibres are apparently relevant to the improvement of fibrefibre bonding. The strength properties of paper made from modified fibres are greatly improved, but the introduction of carboxyl groups also increases the swelling of the fibres. A large fibre surface area provided higher fibre surface charge and more areas for fibrefibre contacts and, subsequently, more number of bonds is developed between fibres. The regression equation agrees excellently with the model predicted data with regression coefficient close to unity. The statistical predictions are claimed to be very good and reproducible with a reasonable degree of accuracy permitted in engineering estimates. Therefore the statistical regression can be used reliably for analysis purposes. The experimental results also showed strong linear correlations between the surface charge and the hand sheets properties.

Table 3:
The experimental value and model predicted values a
$Y = -0.085 X + 450, R^2 = 0.94$

Surface charge (Y) (Experimental data)	Freeness (X)	Model predicted data (Y)	% Error
9.95	430	9.51	4.47
13.65	390	13.31	2.46
17.04	340	18.07	-6.04
24.14	270	24.73	-2.44
29.87	225	29.02	2.84

Table 2:
Relationships of fibre surface charge, specific surface area and specific volume, WRV, tensile index and Canadian standard freeness of pulps.

S. No.	Parameters	Functions	Regression equations	Regression coefficients,R^2
1.	Freeness, ml CSF	Revolutions, No.	y = -0.0850x + 450	0.9439
2.	Surface charge, μeq/g	Freeness, ml CSF	y = -0.095x + 50.44	0.9905
3.	Sp. Surface area, m^2/g	Freeness, ml CSF	y = -0.006x + 5.128	0.9376
4.	Sp. volume (v), cm^3/g	Freeness, ml CSF	y = -0.008x + 6.694	0.9699
5.	WRV, %	Freeness, ml CSF	y = -0.547x + 406.5	0.9424
6.	Tensile index,N.m/g	Freeness, ml CSF	y = -0.059x + 71.94	0.9816
7.	Tensile index, N.m/g	Surface charge	y = 0.615x + 40.725	0.9700

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