

Monitoring the pH and conductivity of dielectric pulps using the ISO and IEC Standard Guidelines

J.M.F. NOGUEIRA, M. FREITAS, AND M. GONÇALVES

ABSTRACT: Regression lines indicated good correlations, with confidence limits at 95%, when comparing the currently used standard guidelines to evaluate the pH (ISO 6588 and IEC 60641-2) and conductivity (ISO 6587 and IEC 60641-2) of kraft pulps. For pH measurements, these standard guidelines resulted in similar results, whereas correspondingly different conductivity values resulted due to the different sample amounts used in the standards. The proper conversion of conductivity values is of great importance, for example, in the case of dielectric pulps ($6 < \text{pH} < 8$; conductivity $< 3.5 \text{ mS/m}$) for research and commercial purposes.

Application: This study will help in comparing pulp qualities measured by different standards.

The growing interest in dielectric pulps, such as used in insulating materials, has put new demands on the worldwide pulp and paper industry to improve pulp washing processes and monitor parameters such as pH and conductivity (γ), especially if dielectric characteristics are required ($6 < \text{pH} < 8$; $\gamma < 3.5 \text{ mS/m}$) [1,2].

International procedures to assess the pH and conductivity parameters of kraft pulps follow standard guidelines from the International Organization for Standardization (ISO) and the International Electrotechnical Commission (IEC). In order to control such parameters in the pulps produced, the analytical departments of mills use the ISO 6588 and IEC 60641-2 standard guidelines to evaluate pH, and the ISO 6587 and IEC 60641-2 standard guidelines to evaluate conductivity [3-5].

IEC standard guidelines are most often used to test materials for electrical purposes, whereas ISO standard guidelines are adopted to test materials having a wide range of applications. Due to the use of different standard guidelines, there are occasionally misunderstandings in the international market in relation to the pulp characteristic requirements and ambiguous data.

This work compares by regression lines the pH and conductivity measurements from kraft pulp samples, using the ISO 6588, ISO 6587, and IEC 60641-2 standard guidelines.

EXPERIMENTAL

Materials and reagents: We used 22 samples of several types of pine pulp obtained from the washing process of the Portucel Tejo SA (V.V. Ródão-Portugal)

kraft mill. The conductivity of the distilled water used for the analytical procedures was below 0.1 mS/m .

Procedure. The pH measurements, obtained with a pH meter (Radiometer Copenhagen® VIT 90), conformed to ISO 6588 and IEC 60641-2 standard guidelines [3,5]. We used a conductivity meter (Radiometer Copenhagen® CDM 3) to obtain conductivity measurements following ISO 6587 and IEC 60641-2 standard guidelines [4,5].

RESULTS AND DISCUSSION

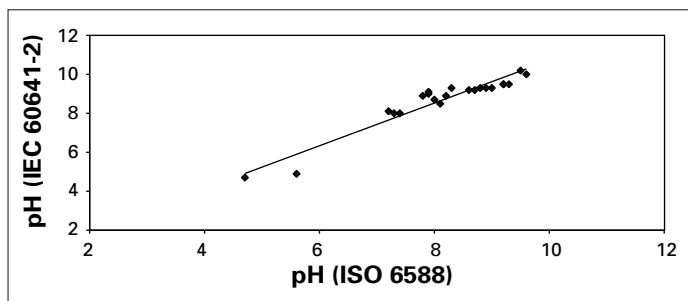
The methodology of the ISO and IEC standard guidelines to measure the pH and conductivity of the kraft pulps seems to be quite similar [3-5]. Both procedures consist of a hot extraction method for 1 h of an accurate weight of pulp (dry basis) with 100 mL of distilled water, followed by the pH and conductivity measurements of the final aqueous extracts at controlled temperature. The main differences between both standard procedures are the amount of sample involved (2 g for ISO and 5 g for IEC) and the temperature control of the measurements (25°C for ISO and 23°C for IEC).

The use of regression lines for data obtained at a suitable analytical range is usually accepted as a way to identify systematic differences when two or more methods are compared [6]. **Figures 1 and 2** show, respectively, the plotted data and regression lines obtained for the pH and conductivity measurements of the aqueous extracts from the 22 kraft pulp samples tested, by the ISO 6588, ISO 6587, and IEC 60641-2 standard guidelines. The results indicated good correlations. **Table I** shows the parameters obtained from the regression lines. The s_a and s_b values can

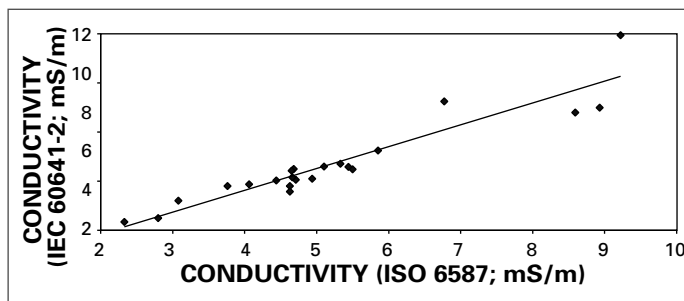
be used to estimate confidence limits for the slope (b) and the intercept (a) in the usual way. Thus, the confidence limits for the slope are given by $b \pm t s_b$, where the t value is taken from the t -distribution at 95% confidence level for $n-2$ degrees of freedom [6]. Similarly, the confidence limits for the intercept are given by $a \pm t s_a$. The correlation coefficient (r) is of less importance in the present context than the establishment of confidence limits for the slope and intercept. A correlation coefficient near one suggests good precision for both standard guidelines.

The pH regression line of Fig. 1 shows an ideal situation ($a \rightarrow 0$; $b \rightarrow 1$ and $r \rightarrow 1$), having the slope close to 1 ($b = 1.093246 \pm 0.156524$) and the intercept close to zero ($a = -0.218620 \pm 1.288240$). It seems to prove that both standard guidelines (ISO 6588 and IEC 60641-2) are quite similar, showing no evidence for systematic differences between the two sets of results. In spite of the amount of pulp sample required for the IEC 60641-2 standard procedure (5 g) being 2.5 times higher than for the ISO 6588 standard procedure (2 g), the pH is hardly affected by the difference. In fact, the pH is a logarithmic scale, which attenuates small variations in the hydrogenionic concentration.

The conductivity regression line of Fig. 2 presents a slope clearly higher than 1 ($b = 1.777396 \pm 0.309916$) and the intercept close to zero ($a = 0.144506 \pm 1.698320$). As expected, the tendency of the conductivity measured by the IEC 60641-2 standard guideline almost doubles in relation to the ISO 6587 standard guideline, as a consequence of the difference in the amount of pulp sample used in both standard procedures. In this case, the differences in conductivity between



1. Plotted data and regression line obtained by comparison of ISO 6588 and IEC 60641-2 standard guidelines, using the pH data on 22 kraft pulp samples.



2. Plotted data and regression line obtained by comparison of ISO 6587 and IEC 60641-2 standard guidelines, using the conductivity data on 22 kraft pulp samples.

both sets of results can be explained through the ionic species usually sorbed in the pulp fibers, e.g., sodium ion [7], which are proportional to the amount of pulp used in the standard procedures. Thus, from the conductivity data, systematic differences are seen between both sets of results.

In short, the regression lines seem to show good correlation parameters with confidence limits at 95% level for 20 degrees of freedom. The present approach seems to be a useful guide in order to convert the pH and conductivity data of the kraft pulps obtained by the ISO into IEC standard guidelines and vice versa, solving particular dubiousness inside the international market and in future research and development projects, especially for dielectric ($6 < \text{pH} < 8$; $\gamma < 3.5 \text{ mS/m}$) pulps.

CONCLUSIONS

In this study, we used regression lines to compare current standard guidelines for evaluating the pH and conductivity of kraft pulps. From the regression parameters obtained, the standard guidelines for pH and conductivity measurements showed good correlations, with confidence limits at 95% level for 20 degrees of freedom.

For pH measurements, the ISO 6588 and IEC 60641-2 standard guidelines seemed to be quite similar. For conductivity measurements, the IEC 60641-2 standard guideline almost doubled in relation to ISO 6587 standard guideline, as expected, due the different amount of sample required in both procedures.

This present contribution seems to be a useful guide in order to convert the pH and conductivity data obtained from the kraft pulp samples by the ISO into IEC standard guidelines and vice versa. **TJ**

ACKNOWLEDGEMENT

The authors wish to thank Portucel Tejo SA for the pulp samples and technical assistance.

	STANDARD GUIDELINES	
	ISO 6588 vs. IEC 60641-2 (pH data)	ISO 6587 vs. IEC 60641-2 (Conductivity data)
Correlation coefficient (<i>r</i>)	0.956136	0.936912
Intercept (<i>a</i>)	-0.218620	0.144506
Slope (<i>b</i>)	1.093246	1.777396
Statistic $S_{y/x}$	0.413976	1.230745
Standard deviation for the intercept (s_a)	0.616383	0.812593
Standard deviation for the slope (s_b)	0.074892	0.148285
Degrees of freedom (<i>n</i> -2)	20	20
Confidence limits in intercept (95%)	-0.218620 ± 1.288240	0.144506 ± 1.698320
Confidence limits in slope (95%)	1.093246 ± 0.156524	1.777396 ± 0.309916

1. Parameters obtained of the regression lines (Fig. 1 and Fig. 2) and confidence limits for the standard guidelines comparison, using the pH and conductivity data on 22 kraft pulp samples.

LITERATURE CITED

1. Crotofino, R.H., Poirier, N.A. and Trinh, D.T., *Tappi J.* 70(6): 95(1987).
2. Lundqvist, S.O., *Pulp Paper Can.* 86(11): T325(1985).
3. ISO 6588, *Paper, board and pulps - Determination of pH of aqueous extracts* (1981).
4. ISO 6587, *Paper, board and pulps - Determination of conductivity of aqueous extracts* (1992).
5. IEC 60641-2, *Specification for press-board and presspaper for electrical purposes. Part 2: Methods of test* (1979).
6. Miller, J.C., "Statistics for Analytical Chemistry", Wiley, New York, USA, (1984).
7. Rosen, A., *Tappi J.* 58(9): 156(1975).

Received: June 22, 2002
Revised: August 19, 2002
Accepted: August 25, 2002

This paper is also published on TAPPI's web site <www.tappi.org> and summarized in the March *Solutions! for People, Processes and Paper* magazine (Vol. 86 No. 3)

Nogueira is assistant professor and Freitas is a PhD student at Chemistry and Biochemistry Department of University of Lisbon, Campo Grande Ed. C8, 1749-016 Lisbon, Portugal; Gonçalves

is the manager of the Process Study and Control Department of Portucel Tejo SA, Centro Fabril de Ródão, 6030-223 Vila Velha de Ródão, Portugal. Email Nogueira at nogueira@fc.ul.pt



Nogueira



Freitas



Gonçalves