Operator Sensitivity in Testing Oil Absorbency of Paper

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ABSTRACT

The penetration of oily substances into paper is important in many grades of paper, for example, the papers to be printed with oil based inks, papers to be coated with paraffin, or papers to be used in contact with greasy foods. The available methods for evaluation of oil absorbency of paper are mostly subjective in nature and suffer from high variability of test results. We have evaluated material and operator variability for the castor oil penetration test method which is considered most appropriate for such applications. The experiments reveal that the values obtained from this test are highly operator sensitive. The test can be gainfully used for quality control purpose within a mill when conducted by one operator but, it has limited value for comparison of results obtained by different operators in different laboratories.

Key words: Oil absorbency, Castor oil penetration, Variability, Operator sensitivity, Repeatability, Reproducibility

INTRODUCTION

Paper is a versatile material; it provides its services in diverse end uses, sometimes in the form as produced in paper mills, but mostly after passing through some converting operations. In spite of development of a large number of methods and instruments for testing different attributes, paper defies its complete characterization.

The Bureau of Indian Standards (1) brings out minimum quality specifications for a number of grades of paper in order to safeguard the interest of the consumer. Often, even after lengthy debates, experts find it difficult to determine the set of necessary and sufficient properties for inclusion in the specification of a particular grade of paper.

In certain grades the conversion operation requires paper to be brought in contact with oily materials, for example, printing inks. A pertinent question asked is whether values of some oil penetration test should be included in the specifications of such papers or not. If yes, what would be the suitable numerical values and how accurately these values could be determined. We conducted an experimental study to find some answers to these questions using two grades of paper: 1) newsprint meant for printing on high-speed offset presses, and 2) carbonizing base paper meant for coating by oil based formulations. We used the castor oil penetration test method which is considered most appropriate for such applications. The experiments reveal that the values obtained from this test are highly operator sensitive. The test can be gainfully used for quality control purpose within a mill when conducted by one operator but, it has limited value for comparison of results obtained by different operators in different laboratories.

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BACKGROUND

Paper is required to resist, during converting operations and end uses, a variety of penetrants gaseous and liquids. The liquid penetrants may be aqueous liquids or oils and greases. The penetration of oily substances into paper is important in many grades of paper, for example, the papers to be printed with oil based inks, papers to be coated with paraffin, or papers to be used in contact with greasy foods. Different grades of paper require resisting oil penetration to different extents. For example, on one extreme are barrier papers that should be completely impervious to oil, and on the other extreme are the absorbent grades of paper that should readily absorb oily penetrants. An example of the latter grade is absorbing kraft paper requiring saturation with a resin. Between these two extremes, there are many grades, where an intermediate absorbency is desired. For example, printing papers should neither be too much absorbent nor completely resistant to oils. A good printing paper should have enough ability to absorb ink so that the ink is quickly immobilized into the paper and dried in fast printing presses. At the same time, the paper should be sufficiently resistant so that the ink does not penetrate deeper into the paper with very little of it remaining on the surface. Another associated effect of excessive absorbency is that the oil of the ink moves into the paper leaving the pigment behind causing a reduction in opacity of the paper. Thus, a high oil absorbency will mean a poor printing result indicated by a low print density- a low print contrast, a high print through, and a high ink consumption.

Fundamental paper characteristics that would explain the paper-oil interactions completely are difficult to define and measure. Presently, the oil resistance of paper is generally measured by the time required for an oily test liquid to penetrate through the paper. For best results the paper should be tested against the penetrant material with which it has to come in contact during its use, but for the purpose of wider applicability of the test results, the type of oil and the method of its application are carefully standardized. For barrier papers (e.g., greaseproof, glassine, and vegetable parchment) where oil penetration is slow, turpentine is used to speed up the test. The procedure is described in TAPPI standard T 454 (2). For papers that require resisting penetration of oily materials of high viscosity such as printing inks, the results of turpentine test do not correlate well with their actual performance. Common tests for oil resistance of printing papers are:

1. The castor oil test which measures the time required for a drop of castor oil to penetrate through the sheet; TAPPI T 462 (3), and IS: 10380 1982 (4).

2. The surface oil test in which a drop of

Table-1: Summary of oil absorbency test results

.Castor oil penetration time, seconds								
	Opr 1		Opr 2		Opr 1		Opr 4	
ID	Mean	S. Dev	Mean	S. Dev	Mean	S. Dev	Mean	S. Dev
CP1-A	11.9	1.4	6.8	0.9	8.7	1.2	9.5	1.6
CP1-B	10.7	1.2	7.2	0.8	9.3	1.1	9.8	1.2
CP1-C	9.6	1.3	6.4	0.6	6.2	0.9	7.0	1.6
CP1-D	10.0	1.1	6.8	0.6	7.9	0.7	8.9	1.2
CP1-E	9.7	1.0	6.1	0.8	9.1	0.5	8.6	0.7
CP2-A	8.2	1.2	5.3	0.9	4.3	0.7	4.4	0.6
CP2-B	9.4	0.9	5.1	0.5	4.8	0.6	5.4	0.5
CP2-C	9.1	0.9	4.9	0.4	4.7	0.4	6.6	0.9
CP2-D	7.3	1.4	5.8	0.6	5.1	0.5	6.0	1.1
CP2-E	8.5	1.2	6.0	0.8	5.2	0.6	5.2	0.7
CP1*	10.4	1.2	6.7	0.7	8.2	0.9	8.8	1.3
CP2*	8.5	1.1	5.4	0.7	4.8	0.6	5.5	0.8
NP1	46.8	5.5	35.3	3.4	37.1	4.6	44.2	4.2
NP2	58.3	5.9	44.7	4.9	48.4	5.3	51.4	4.6

* Grand mean and grand standard deviations of CP1-A to CP1-E and CP2-A to CP2-E respectively.

linseed oil is placed on the surface of a rubber covered roller and is spread on the paper into an oblong shape by passing the roller across it. The time required after application of oil onto paper for the sheen (specular reflection at 85) to disappear is measured.

3. The K&N ink stain test; TAPPI RC-19 (5). In this method, an excess of a standardized test ink, named K&N ink after the supplier, which essentially consists of a coloured oil with a white pigment extender, is applied (buttered) to the surface of the paper. The ink is allowed to remain there for a specified contact time after which the excess of it is removed from the surface by wiping with a cleaning tissue. The intensity and appearance of the resulting stain are assessed which give a measure of the extent of absorption of the oil by the paper.

One or more of these methods are used in the industry for quality control purposes and in monitoring technical developments. The castor oil penetration test has been adopted as an official test method by TAPPI (T 462). This method has also been accepted as a standard method by the Bureau of Indian Standards (IS: 10380 - 1982). A test result is a measurement of the time required for a drop of castor oil applied on the surface of a test specimen to produce a uniform translucent spot on the other side of the specimen by permeation through it. The type of the castor oil used, the amount of oil in the drop, and the method of application of the drop onto the paper surface are standardized and controlled during the test. Since the determination of the end point, i.e., the time to achieve uniform translucency, is of subjective nature, a considerable variability exists in the test value obtained by different observers.

EXPERIMENTAL

Samples of commercial newsprint and one-time carbonizing tissues were used in the present study. Two newsprint samples, NP1 and NP2, were obtained from a paper trader who drew them from the supplies of two different manufacturers. Ten samples of base paper for one time carbon paper were obtained from two different manufacturers. The samples CP1-A to CP1-E were drawn from five different batches of the same quality carbonizing base paper made by one manufacturer, and the samples CP2-A to CP2-E were drawn from five batches of carbonizing base paper made by the other manufacturer. Both the qualities of paper were known to give satisfactory performance during carbonizing process. For determination of castor-oil absorbency of paper, the testing procedure as outlined in IS:10380 - 1982 was followed. A wooden box apparatus was fabricated with details consistent with the standard and the medical grade castor oil was used for the experiments.

Since the determination of the end point in the caster oil penetration test is of subjective nature, the samples were tested by four observers (operators). The observers were chosen from those familiar with the paper testing work. Each observer was requested to carry out the test on five specimens for each side of each sample and record the values of time for oil penetration in seconds.

DISCUSSION OF RESULTS

The average and the standard deviation of the five values of oil penetration times recorded for each side by an operator were calculated. We did not notice much difference in the oil penetration times for the two sides of any sample. We, therefore, combined the test results for the two sides and calculated average and standard deviation of ten observations for each sample as shown in Table-1.

For determining the variability in test results in numerical terms, we analyzed the experimental data statistically using the principles outlined in Tappi standard practice T 1200 sp-00 (2c). The variations in test results have been expressed in terms of 'material variability' and 'operator variability'.

We have defined the material variability on the lines of Tappi repeatability precision statement. It is an estimate of the maximum difference between two test results with 95% confidence for a sample, when the test results are obtained under the same testing conditions. The meaning of the same conditions covers same homogeneous source of material, same laboratory, same operator, same apparatus, same environment and testing conditions as far as possible. Similarly, we have defined the operator variability on the lines of Tappi reproducibility. It is an estimate of the maximum difference between two test results with 95% confidence, when the test results are obtained under the same testing conditions except that the operators are different. The 95% confidence level is a generally accepted confidence for precision estimations.

Let p be the number of operators, n be

the number of test results recorded by an operator for a sample, and x's be the values of test results. The following statistical parameters have been calculated for each sample:

Material mean: $\bar{x} = \sum x / n$

Material standard deviation: $\sigma_x = \sqrt{\sum (x - \bar{x}) / (n - 1)}$

Grand material mean: $\bar{\bar{x}} = \sum \bar{x} / p$

Grand material standard deviation: $\sigma_{\bar{x}} = \sqrt{\sum (\bar{x} - \bar{\bar{x}})^2 / (p - 1)}$

Pooled material standard deviation: $\sigma_p = \sqrt{\sum \sigma_x^2 / p}$

Material variability: $r = 2.77\,\sigma_p$

Material variability ratio: $\%r = 100(r/\bar{\bar{x}})$

Operator standard deviation: $\sigma_R = \sqrt{\sigma_{\bar{x}}^2 + \sigma_p^2 (n - 1)/n}$

Operator variability: $R = 2.77\,\sigma_R$

Operator variability ratio: $\%R = 100(R/\bar{\bar{x}})$

For calculating material variability, the pooled material standard deviation is multiplied by $1.96\sqrt{2}(= 2.77)$. The factor 1.96 is the confidence coefficient at 95% confidence level for the normal distribution of errors (usual assumption). The $\sqrt{2}$ is chosen because the variability strictly applies to differences between any two test results. The operator variability is calculated in a similar manner.

Table-2: Summary of calculations for material and operator variability for each sample

ID	Grand Mean, $\bar{\bar{x}}$	Grand STd. $\sigma_{\bar{x}}$	Pooled S. Dev σ_p	Opr s. Dev, σ_R	Material variability r	Operator variability R	Material variability Ratio (%)	Operator variability Ratio (%)
CP1-A	9.23	2.11	1.29	2.44	3.57	6.76	39	73
CP1-B	9.25	1.48	1.08	1.80	2.98	4.99	32	54
CP1-C	7.30	1.57	1.18	1.93	3.26	5.34	45	73
CP1-D	8.40	1.37	0.93	1.63	2.57	4.51	31	54
CP1-E	8.38	1.58	0.76	1.74	2.11	4.82	25	58
CP2-A	5.55	1.82	0.90	2.01	2.48	5.57	45	100
CP2-B	6.18	2.16	0.64	2.25	1.77	6.23	29	101
CP2-C	6.33	2.04	0.71	2.14	1.96	5.94	31	94
CP2-D	6.05	0.92	0.97	1.30	2.70	3.61	45	60
CP2-E	6.23	1.56	0.88	1.77	2.44	4.91	39	79
CP1*	8.51	1.53	1.06	1.83	2.94	5.08	35	60
CP2*	6.07	1.65	0.83	1.83	2.30	5.07	38	84
NP1	40.85	5.52	4.49	6.97	12.43	19.32	30	47
NP2	50.70	5.76	5.20	7.58	14.40	21.00	28	41

* As explained in Table-1

Fig.1a: Material and Operator variability for samples of carbonizing base paper.

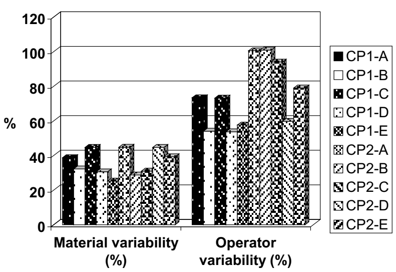

Fig.1b: Material and Operator variability for carbonizing base papers (grand mean of five batches) and newsprints.

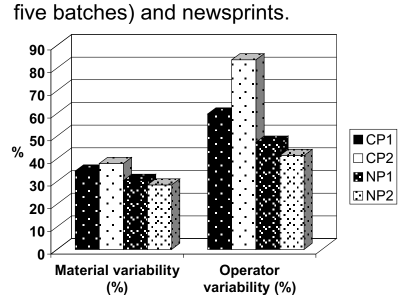

Table-2 shows the values of various statistical parameters and the material and operator variability for each sample. Figs. 1a and 1b show this variability graphically. There was a large material variability (between 30 and 40%) for all the samples included in the tests. The operator variability was very large, about 100% for some samples. The operator variability appears dependent on the magnitude of the test results. The operator variability was the greatest for the sample CP2 that has the lowest mean oil penetration time, and was low for the newsprint sample that had high oil penetration time values.

Consistency evaluation

The h-statistics and k-statistics were used to determine if any of the experimental data showed inconsistent values, general trends or unusual patterns.

Cell difference: $d = \bar{x} - \bar{\bar{x}}$

Mean consistency statistic: $h = d / \sigma_{\bar{x}}$

Standard deviation consistency statistic: $k = \sigma_x d / \sigma_p$

Table-3: Summary of h-statistics and k-statistics for different operators for each sample

Sample ID	h-statistics				k-statistics			
	Opr 1	Opr 2	Opr 3	Opr 4	Opr 1	Opr 2	Opr 3	Opr 4
CP1-A	1.27	-1.15	-0.25	0.13	1.05	0.70	0.93	1.24
CP1-B	0.98	-1.38	0.03	0.37	1.08	0.74	1.02	1.11
CP1-C	1.46	-0.57	-0.70	-0.19	1.14	0.51	0.77	1.36
CP1-D	1.17	-1.17	-0.37	0.37	1.17	0.65	0.76	1.28
CP1-E	0.84	-1.44	0.46	0.14	1.27	1.05	0.66	0.92
CP2-A	1.45	-0.14	-0.69	-0.63	1.35	1.05	0.75	0.71
CP2-B	1.49	-0.50	-0.64	-0.36	1.36	0.78	0.91	0.84
CP2-C	1.36	-0.70	-0.80	0.14	1.32	0.61	0.52	1.27
CP2-D	1.36	-0.27	-1.03	-0.05	1.44	0.62	0.53	1.13
CP2-E	1.46	-0.14	-0.66	-0.66	1.41	0.94	0.71	0.79
CP1*	1.22	-1.21	-0.18	0.16	1.12	0.71	0.86	1.23
CP2*	1.47	-0.39	-0.75	-0.33	1.38	0.83	0.68	0.97
NP1	1.08	-1.01	-0.68	0.61	1.23	0.76	1.02	0.94
NP2	1.32	-1.04	-0.40	0.12	1.14	0.94	1.02	0.88

* As explained in Table-1

Table-3 shows the calculated values of h and k for an operator for each sample. Critical values of h and k were obtained from Table-6 of Tappi Standard Practice (2c). At 0.5% significance level, the critical values of h = 1.49, and the critical value of k = 1.47 for 4 operators and 10 replicate measureme - -nts.

For assuming experimental data to be consistent, the h and k values should be less than the critical values; h values in excess of critical value indicate excessive variation of the data from the grand mean, and k values in excess of

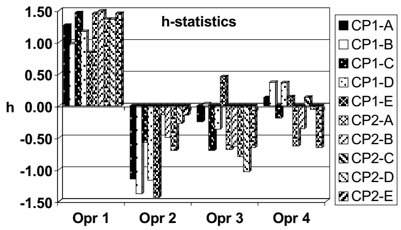

Fig.2a: h-values for different operators for samples of carbonizing base paper.

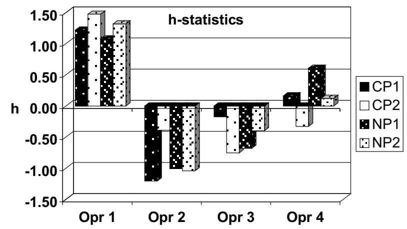

Fig.2b: h-values for different operators for carbonizing base papers (grand mean of five batches) and newsprints.

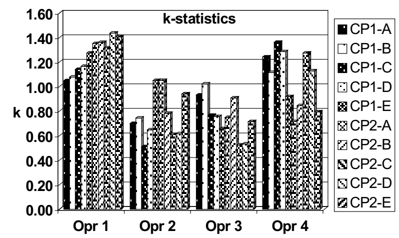

Fig.3a: k-values for different operators for samples of carbonizing base paper.

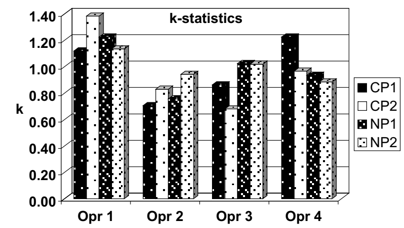

Fig.3b: k-values for different operators for carbonizing base papers (grand mean of five batches) and newsprints.

the critical value indicate excessive variation in the data from the pooled material standard deviation. Figs. 2 and 3 graphically show the k and h values given in Table-3. Although the h and k values were within the critical limits for all the samples and operators, there were some visible inconsistencies in the judgement of different operators. The test results of operator 1 were greater than the grand mean for all samples, while the results of operator 2 were always less than the grand mean. The results of operator 4 were closest to the mean; greater than the grand mean for some samples and less for the others. Further, the test results of operator 1 deviated most from others indicated by the greatest values of h and k in its case.

CONCLUSIONS

1. The caster oil penetration test suffers from large material and operator variability.

2. The determination of the end point in the test is not well defined and it is operative sensitive.

3. The operator variability is high for highly absorbing papers and low for less absorbing papers.

4. In making quality specifications for any grade of paper, it is essential that the properties included in the specifications should be capable of being measured within a narrow range in different laboratories by different operators. Because of high operator variability, inclusion of a meaningful narrow range of castor oil penetration test values appears difficult.

5. In spite of large variability, oil absorption tests are very important in characterization of many grades of paper. The castor oil penetration test results are useful in comparing paper samples evaluated by a single operator.

6. The agreement between operators can, perhaps, be increased by exchange of test results among them.

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