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Use of Microtome Sections to Determine the Effect of Retention Aid on Filler Distribution

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USE OF MICROTOME SECTIONS TO DETERMINE THE EFFECT OF RETENTION AID ON FILLER DISTRIBUTION

by

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Two-sidedness of paper is an all too common problem in the paper industry. Most of the undesirable results are due to the uneven distribution of filler in the sheet. An accurate, reproduceable, method is needed to study this distribution gradient in order to find the effect of certain variables on this distribution. A lthough the following method has been used before, its reproducibility has not been demonstrated to any great extent, nor has much work been done as to the effect of variables on the distribution. In this study, the reproducibility of this method is demonstrated and the effect of retention aid is studied.

Previous reports of paper sectioning to determine filler distribution have been with both approximate and more exacting techniques and with some study of variables affecting this distribution. Since color, finish, and brightness two-sidedness are usually a result of the different concentrations of filler on the felt and wire sides of the sheet, any way of leveling out this distribution would be of great value to the papermaker.

Initial work in sectioning was done by Schilde (1) who demonstrated the fact that paper was two-sided by scraping off layers with a razor blade and ashing the remains. Doing his work in 1930, he said that two-sidedness was due to the action of the wire and suction boxes. He found that the difference between wire side and felt side ash content increases with machine speed and filler, and decreased with higher basis weight. However, his work was done on a slow machine and much of it cannot be directly applied to today's faster machines.

In more recent work, Hansen (2) in 1951 used carborundum paper to grind off certain portions of the sheet and ash the remains. However, he too worked in a slow machine (170-265 f.p.m.) and found that at this speed, the addition of Sveen glue had no effect on the distribution. He found, as others before, that the felt side ash was about twice as high as the wire side. He further found that the washing action of table rolls has no effect at that slow speed. Thus, his work cannot be used to explain the two-sidedness on faster machines, or how certain factors would affect it.

In a much more thorough study at 800-1000f.p.m., Underhay (3) used gummed Kraft paper to split his sample into four sections to determine the filler distribution through the sheet. He found that no two-sidedness was apparent by using an Ingersoll Glarimeter or Bekk Smoothness Tester. He went on to show that the removal of fines preferentially from the bottom of the sheet is a result of the disturbances and washing of the underside of the sheet during its passage over the table rolls. He did some work with hand sheets and found that the only way two-sidedness comparable to that on the machine could be obtained was by vibrating the entire sheet mold during draining at 700 vibrations a minute.

Several studies have been published on the use of the microtome, initially started by Browning and Isenberg (4). They did no work with factors determining distribution, but simply showed the rapid decrease of filler content, from felt to wire side. They did find however, that it was diffibult to section coated papers because the microtome knife tended to cut off the whole coated layer instead of cutting through it. The method of sectioning used by Browning and Isenberg is the same as that used in this study which will be explained later.

H. Mack and B. Kleu (5) followed Browning and Isenberg's work using a microtome and further demonstrated the uneven distribution of filler. In comparing the distribution in two different sheets with 10% and 20% ash, it was found that the higher ash sheet has a difference in ash from felt to wire side of 17% while the other, a change of only 6%.

A new non-quantitative method developed by A. Lehtinen (6) made use of microtome with an angle indicator that produced slant sections through the sheet. These sections were stained and examined under a microscope to visually estimate the concentration of filler through the sheet, while also noting wire mark and ink penetration of printing. A new method of sectioning was developed by Parker and Mih (7). Water-soaked paper was fed between two chilled rolls, freezing the surface of the paper to the rolls. The rotation of the rolls divided the sheet in half. This enabled microscopic investigations of large samples of paper. It was particularly useful for observing ink penetration, formation and specific filtration of various layers of the sheet, besides being able to ash each layer with the use of a common analytical balance.

Many of these studies indicated that table rolls were the main cause of two-sidedness. This is a result of the carrying up of water by the roll into the nip between the roll and wire, distrubing the initial formation of the web.

Thus the purpose of this paper is to further demonstrate the use of microtome sections, the reproducibility and accuracy of the method, and a beginning study into the effect of retention aid on the distribution of filler.

PROCEDURE FOR SECTIONING

An American Optical Sliding Microtome is used for sectioning the paper. All sectionings and weighings are done in a constant himidity room at 50%R.H. and 72°F. A microbalance is used for weighing the crucibles and samples. Size ooooo crucibles are used to ash the sections in, with matching covers and pans.

To hold the paper in position, small wooden blocks (10x 15x20mm-grain long) cut out of soft pine are used. Once block is placed in a corner of the sample holder on the microtome and a shorter block placed in the opposite corner to be glued in place permanently. This gives a more uniform pressure on the blocks that are taken out and replaced during the sectioning.

After the block is fastened firmly in the sample holder in a vertical position, the entire assembly is raised by means of the crank on the back side, until the knife just touches the block. The depth of cut is set with the adjusting screw at the base of the sample holder. Sections of 15 micra are taken. Then the block is leveled by taking equal 15μ sections until the surface is smooth. Twenty blocks are leveled in this manner.

Small pieces of paper (about 7x12mm) are cut from the sample and glued to the level surface of each block by applying Duco cement with the finger onto the paper. Ten samples are glued wire side down and ten felt side down in this same way. Next, small thick glass plates, about one inch square, are pressed on top of the paper, and allowed to rest there for one hour to keep the paper level and allow the glue to dry. Too much glue should not be applied to the sample or it will penetrate too far through the sheet and throw off the ash determinations. It is also very important that the paper be level or slant sections instead of aprallel sections will result.

After drying, the block and sample are replaced in exactly the same position as at first, and then loweredjust slightly below the knife. The specimen is raised by 5μ increments until the knife just grazes the surface of the paper. The sample is then raised 15μ at a time and four sections taken from the outer side. Only four sections are taken from each side since the glue seems to penetrate about half-way through the sheet. The paper itself is from six to seven sections thick for 50 lb. and 60 lb. basis weight.

One problem in sectioning should be noted here. In taking the first section from each side, a section much larger than 15μ was always obtained, while the next layer was very small. This was probably due to the strength of the surface from starch penetration. To correct this, when the sample was raised for the first section, the crank was turned back slightly, thus obtaining a sample more closely equal to the following ones in thickness.

The sections are removed from the knife with a camel's hair brush into a micro-crucible that has been previously weighed on the microbalance. Each layer is put into a separate crucible and the same respective layer from each sample is accumulated in the same crucible. After all sections are cut, the sections are allowed to come to equilibrium weight and then re-weighed on the microbalance. The crucibles and samples, with covers on, are placed in a muffle furnace at 1500-1700°F for one-half hour. They are removed after this period and allowed to come to equilibrium weight in the constant humidity room and then weighed to determine the weight of remaining ash. The percent ash of each layer is plotted versus the average distance from the felt side in microns.

It is recommended that a more viscous glue be tried in any future work since the Duco cement seemed to penetrate very easily.

DETERMINATION OF FILLER DISTRIBUTION

A standard offset grade of paper was used for sectioning. This paper was run on a standard Fourdrinier at 850f.p.m. Samples were taken from 60 lb. (25x38-500) paper at retention aid additions of .29 lb. and .58 lb. retention aid per ton of paper. The samples were about 115μ thick. The results are shown in Table I and fig. 1. Sections were also taken on 50 lb. (25x38-500) samples with the data shown in Table I and fig. 2. This paper was about 100μ thick. Duplicate runs were made on several of the 50 lb. samples to determine the reproducibility of the method. It was found that the duplicate runs agreed withing 0.2% except for a few slightly larger variations. The samples were also ashed whole to determine the total filler content. Fig. 3 shows the relation between the various runs when plotted together.

DISCUSSION AND CONCLUSIONS

The results for the 60 lb. paper plotted in fig. 1 present an interesting distribution. The graphs show that the top 2/3 of the sheet was practically constant in filler content in the .58 lb./ton sheet, while the distrubance caused by the table roll action seems to reach further in the sheet at the lower addition. This would be expected since with less floculation there would be smaller clusters of filler particles which would be washed out more easily. However, it can probably be said that over this range of concentration, the change in amount of retention aid has no significant effect on the distribution. This can also be seen in Table I where the % ash in the layers differed by no more than 2% in any two respective layers.

The plots for the distribution of the 50 lb. sheets shown in fig. 2 present us with quite a different picture. The extensive undisturbed layer that was so evident in 60 lb. sample does not take up as great a portion of the sheet in this case. Actually, the ash starts decreasing almost immediately from the felt side. The total difference in ash content from felt to wire side is also much greater than in the 60 lb. paper. This would be expected since the washing action would effect a larger portion of the 50 lb. than 60 lb. because of the difference in caliper.

In comparing the 50 lb. curves with varying retention aid, again no significant change in the shape of the curve is observed. A slightly faster decrease in filler content can be seen at the lower addition. This is also shown in Table I, but probably would not be significant. Here again the difference in ash was not much over 2% for any layer in the various sheets.

While the three plots of samples containing retention aid have the same general shape, the results for the sample with no retention aid has quite a different form. The top two thirds of the sheet is virtually undisturbed while the wire side has a very sudden drop off in filler content. Although it is unfortunate that the sample has a lower ash content than the others, it can be reasonably said that while the felt side would have a higher filler content with a higher ash, the shape of the curve would most likely remain the same.

It is interesting to note on the 50 lb. curves with retention aid, that the fourth point from the felt side fell below the curve in each instant. This is probably due to the penetration of glue into this layer, which would give a larger weight before ashing, but the same weight after ashing. This most likely happened because the wire side was glued down, and the glue would penetrate farther from that side than from the felt side because it is more open and has a lower concentration of clay.

It is known that smaller particles will filter through a web easier than larger particles. This probably can explain the results obtained. With little floculation taking place, except electro-static and alum floculation, the filler in the upper part of the sheet would be expected to naturally filter through as the water drains. With only filtering evidently taking place in the upper half of the sheet, a fairly constant amount would be expected to remain throughout that portion depending on the inter-fiber openings. Near the wire side, the effects of the washing action of the table rolls is present, disturbing the natural filtering action. With retention aid, the heavily floculated particles will not filter through very readily, and will ten to lay on top of the sheet.

This method has a high degree of accuracy and reproducibility, which is shown from the agreement of the duplicate runs. It can be used with confidence without having to do checks, since the procedure is quite lengthy and tedious.

It should be recognized that this has not been an exhaustive study, but merely a beginning investigation into the effect of retention aid on filler distribution. From these limited samples, we can reasonably say that two-sidedness can be reduced by not using retention aid. This has been the case in may mills where retention aid was taken out to reduce color two-sidedness. This occurs because of the uneven distribution of filler dye and/or fiber-dye combinations. Also, in two-side coating, the difference in the amount of coating needed for each side would be less. However, in the few limited cases where printing is only done on one side in uncoated grades, a higher ash content on the felt side would be desirable both for finish and for ink receptivity, thus requiring the use of retention aid. Pick strength would be affected by the use or non-use of retention aid, the former having a deleterious effect. Thus, by knowing the end use of the paper, one can use or not use retention aid to change at least in part, certain attributes such as finish, and wax that would normally be changed by other means. This, then, could give the papermaker another tool by which to control the manufacture of paper.

LITERATURE CITED

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Table I

COMPARISON OF SECTION ASHES

60# OFFSET

13.7% Ash
R. A. - .29#/ton

Felt	16.9%	-	
	16.3	-	
	16.9	-	
	15.5	16.3	
	-	14.7	
	-	10.5	
	-	8.6	Wire

14.4% Ash
R. A.- .58#/ton

Felt	17.1%	-	
	16.2	-	
	16.5	-	
	13.5	15.8	
	-	16.1	
	-	12.5	
	-	10.2	Wire

50# OFFSET

16.8% Ash
R. A. - .58#/ton

Felt	21.4%	-	
	22.0	-	
	20.4	21.4	
	14.9	20.0	
	-	14.0	
	-	9.6	Wire

15.2% Ash
R. A. - .75#/ton

Felt	21.2%	-	
	20.8	-	
	19.5	19.4	
	14.0	18.4	
	-	14.3	
	-	9.3	Wire

13.0% Ash
R. A. - 0

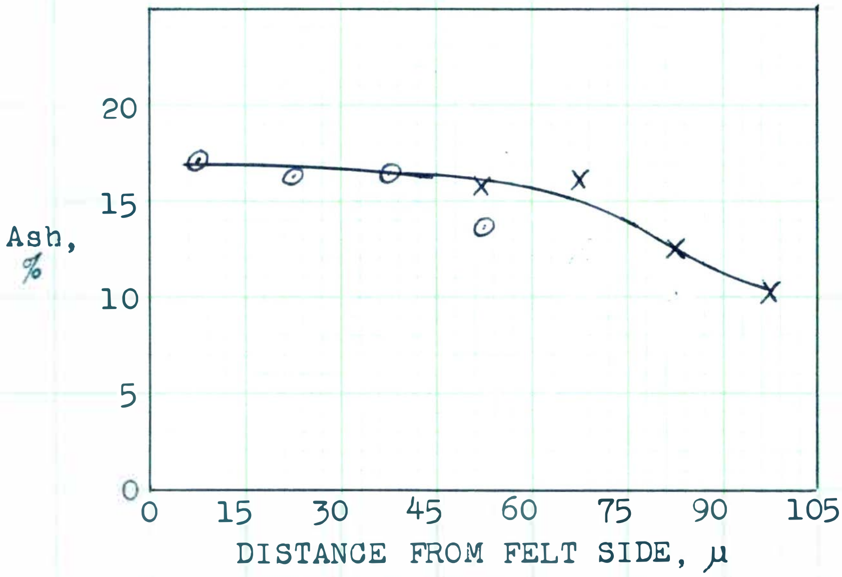
Felt	15.3%	-	
	15.3	-	
	14.8	14.9	
	-	14.7	
	-	10.5	
	-	8.4	Wire

15.5% Ash
R. A. - .29#/ton

Felt	20.0%	-	
	20.1	-	
	18.1	18.5	
	14.5	16.4	
	-	12.1	
	-	8.0	Wire

DISTRIBUTION OF FILLER IN 60# OFFSET SHEETS

R.A. - .58#/ton   14.4% ASH



Section cut from:
⊙ Felt side
× Wire side

R.A. - .29#/ton   13.7% ASH

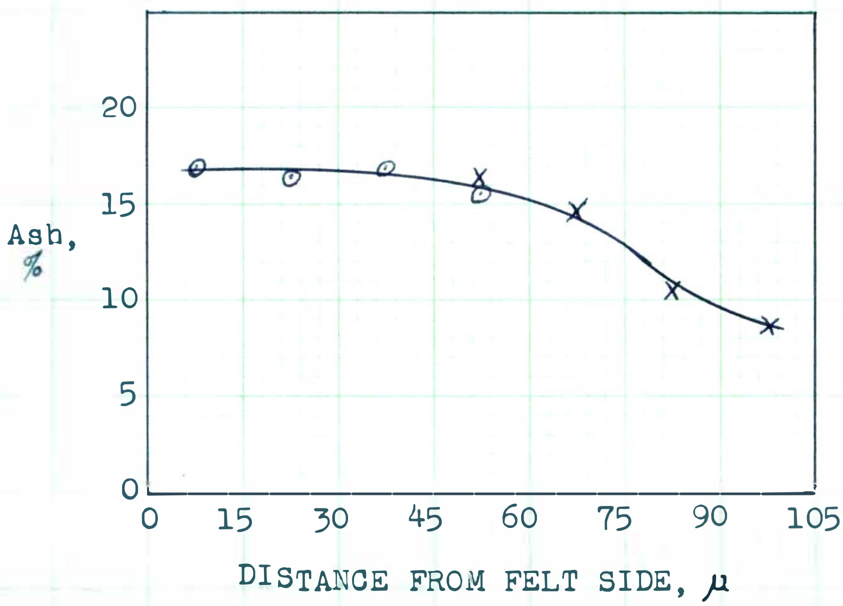


Figure 1

DISTRIBUTION OF FILLER IN 50# OFFSET SHEETS

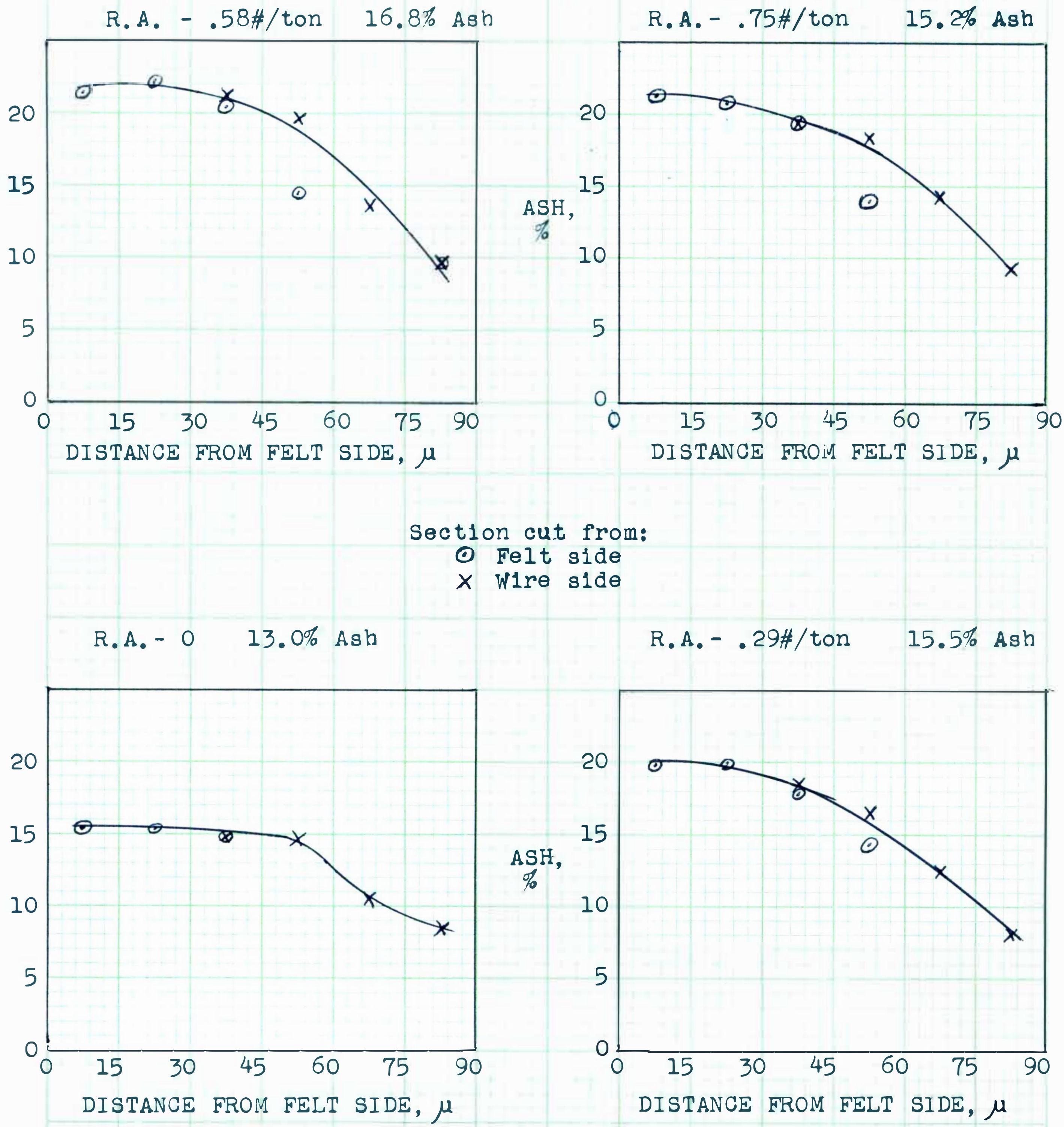
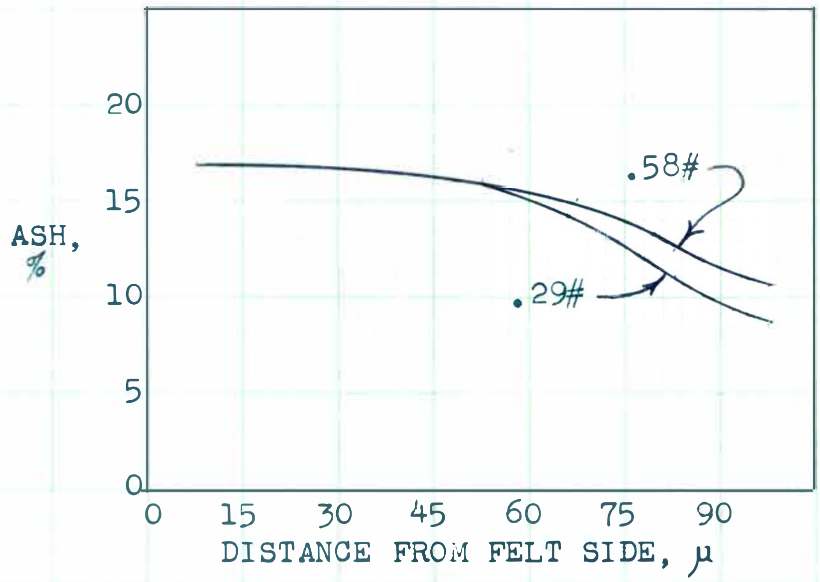


Figure 2

COMPARISON OF DISTRIBUTION WITH DIFFERENT AMOUNTS OF RETENTION AID

60# OFFSET



50# OFFSET

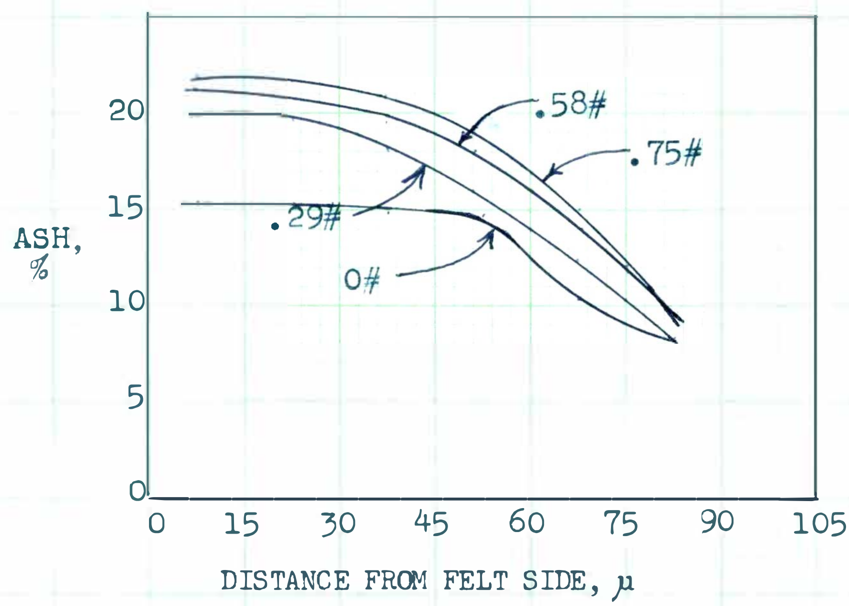


Figure 3