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The Effect of Light Exposure on Sizing Response

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The Effect of Light Exposure on Sizing Response

By

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Submitted to Faculty of Paper Science and Engineering
In Partial Fullfilment of the Requirement for
The Degree of Bachelors of Science
Department of Paper Science and Engineering

Western Michigan University
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ABSTRACT

Sizing is the process by which a chemical additive provides paper with resistance to liquid wetting, penetration, and absorption. A common problem in the paper industry is size reversion, where the paper loses its ability to withstand liquid penetration. This problem has been significantly evident with the use of AKD, and becomes more of a concern as the paper industry shifts towards alkaline papermaking. Many variables have been studied however a direct correlation between the cause and effect of reversion has not been determined. One variable that has not been studied is the effect of light exposure. The objective of this experiment is to determine if light exposure has an effect on sizing by causing reversion.

Handsheets were made and sized with AKD at various addition levels. Samples were then exposed to indoor light conditions. Samples were also exposed to direct UV light using a fade-ometer. Sizing response and brightness were recorded over a fifteen week period. The results indicated that size reversion was evident in all samples exposed to the indoor light and the UV light. The lower sizing levels were most effected, losing more than 95% of the initial sizing level within the first week of exposure. Higher sizing levels were effected to a lesser degree but still indicated the same results. Brightness results indicated that the lower sizing levels showed a slight decrease in value while the higher addition levels gave a net increase in brightness. The decrease is attributed to deterioration of the cellulose while the increase can be due to bleaching of the cellulose.

It is believed that light exposure causes reversion by forming a hydrolysis product of the unbound AKD. The light acts as an energy source to form this hydrolysis product which is a known desizing agent. Reversion is also a function of the deterioration of cellulose, where upon irradiation the chain length of cellulose is decreased and the ester bond between AKD and cellulose is cleaved. Finally, light speeds up the rate of the reverse reaction which is the breaking of the size to cellulose bond.
INTRODUCTION

Sizing is the process by which a chemical additive provides paper with resistance to liquid wetting, penetration, and absorption (1). Size reversion is the event where the paper loses this ability. This problem has been significantly evident in the alkaline papermaking range. Alkyl ketene dimer (AKD) is the premier sizing agent at the alkaline range, followed closely by alkenyl succinic anhydride (ASA). AKD is the most often referenced in size reversion studies, however literature to date does not definitively determine what causes size reversion. As the industry continues to move in the direction of alkaline papermaking, there is a need to study size reversion. Although several recent authors have given their reports on what might cause size reversion, it is this authors opinion that one area has not been studied. That area is the effect of light exposure on sizing response.

This senior thesis is designed to test this authors hypothesis that light does effect the sizing response of paper. This report will give in detail a literature review of the important findings in this area, the experimental design, the significant results, and conclusions of the findings.

ANALYSIS OF LITERATURE

Advantages of Alkaline Papermaking

The production of alkaline paper is increasing. Currently 20% of all paperboard, and 70-80% of all white grades produced in the United States is alkaline. In Europe this figure is closer to 50-60%, and expected to grow (1). Many positive claims have been made for alkaline papermaking. The three most common alkaline sizing agents used are alkenyl succinic anhydrides (ASA), alkyl ketene dimers (AKD), and neutral rosin systems. AKD based sizing agents have been widely used by the paper industry and particularly effective since the early 1950’s (2). The advantages cited for alkaline systems over the traditional acid systems include the following (1):
• Improved sheet strength
• High filler loading that replace expensize fiber
• Reduction in energy consumption
• Increased productivity
• Reduced corrosion
• Increased system closure
• Improved paper stability on aging
• Substitution of calcium carbonate for titanium dioxide.

Although there is great debate over some of these claims, it is without a doubt the last two statements that have significantly prompted the increased use of alkaline papermaking. According to Strutz (3) the stimuli for growth in alkaline papermaking has been the widespread availability of natural ground calcium carbonate. There have also been increased use of on site satellite manufacturers of precipitated calcium carbonate (PCC). This along with the better technology leading to the development of synthetic sizing agents have produced the most successful combination since rosin and alum.

Economic advantages are taken by loading the sheet with this inexpensive filler. The adverse effect is that regardless of the carbonate utilized, the more filler that is added, the lower the sizing response. This has been especially evident in the use of high surface area scalenohedral PCC (4).

Permanence

Librarians and archivists support alkaline papermaking due to the benefit of paper stability upon aging (5). Permanence is the characteristic of paper which determines the extent to which a paper will retain its original properties after aging (6). Papermaker’s alum used in the acid system contributes sulfate ions along with aluminum ions. The sulfate and aluminum ions are free to react with available protons to form weak acids. From this, acid hydrolysis is believed to be one of the chemical reactions that result in the
loss of paper permanence. One commonly accepted method of determining permanence is whether the paper holds its color over time. The color reversion is the general loss of brightness and whiteness. Alkaline papers are superior to acid papers in keeping their properties (1). Brightness reversion and yellowing are common to acid paper and are attributed to the development of chromophoric groups by oxidation (6).

Aging of paper and the study of its permanence has been documented by several authors (7-15). In general the problems surrounding the degradation of cellulosic materials involve chemical, mechanochemical, thermal, photolytic and radiation degradation’s. The conclusion is drawn that the decomposition of cellulose involves mainly hydrolysis of glucosidic links, regardless of paper pH. Intense degradation in alkaline media may be attributed to the action of hydroxyl groups or hydronium ions. Literature also sites that resistance to aging depends on the nature and purity of the raw material. The permanence will be impaired by lignin, hemicellulose, sizing (especially rosin/alum), and high filler content. The thermal and mechanical degradation’s involve free radicals. We also know that cellulose will deteriorate in the presence of light due to the chain length of cellulose being decreased (16). Gray (17) notes that the typical method of determining the paper pH through an extract is not a reliable single factor by which to judge the permanence of paper.

Very few studies have examined the effect of light exposure on paper and sizing response. The loss of brightness and yellowing due to the formation of color bodies are common features of paper aging. This is especially evident in acid made paper and is attributed to the development of chromophoric groups by oxidation (especially carbonyl, phenolic, and guinone groups) (4). Work by Reinhardt (18) has shown that AKD size reduced the light induced yellowing evident with rosin and alum. Graminski and Parks (19) determined that the rate degradation of paper at elevated temperatures depends on the amount of water bond to cellulose and that which is trapped within cell walls. Experiments have also shown that UV light exposure in air causes a photodegradation of
cellulose and hemicellulose. Rearrangement occurs of the degraded cellulose and hemicellulose to form new stable hydrogen bonds during irradiation (20). The report also confirmed that AKD was not affected by light exposure by losing brightness. Roberson noted that the UV light is in fact an important factor in the deterioration of paper. He went on to conclude that the UV light is similar to that of thermal degradation by promoting oxidation (21).

**Size Mechanism with AKD**

Although there has been great debate as to the exact mechanism of AKD, it seems well documented (22-26) that the bond formed is a covalent ester bond with cellulose hydroxyl groups. The preferred reaction in the presence of cellulose is as follows:

\[
\begin{align*}
R & \quad \text{AKD} \\
\text{R-CH}=\text{C-CH-R} & \rightarrow \text{Cellulose-O-C-CH-C-CH}_2\text{-R} \\
\text{O}=\text{C}=\text{O} & \quad \text{Sizing with Cellulose}
\end{align*}
\]

The lactone ring of AKD opens as pH increases above 5.5. The opened ring can then form the speculated ester bond with cellulose to promote sizing. The R units represent the hydrophobic portion of AKD consisting of 16 to 18 carbon atoms. The sizing reaction is strived for during papermaking. However their is a side reaction that can be present. By having anhydride or diketene reactive groups the size will react with hydroxyl groups of water. For this to occur, the conditions must favor the following reaction:

\[
\begin{align*}
\text{R-CH}=\text{C-CH-R} & \rightarrow \text{R-CH}_2\text{-C-H-R} & \rightarrow \text{R-CH}_2\text{-C-CH}_2\text{-R} & + \text{CO}_2 \\
\text{O}=\text{C}=\text{O} & \quad \beta\text{-Keto Acid} & \quad \text{Ketone}
\end{align*}
\]
The hydrolysis is undesirable at any time because it reduces the amount of active AKD you have available to promote sizing. The literature concludes that the ketone is a known desizing agent, and some believe that the ketone will detract from sizing.

Patton (27) announced that as little as 15% of the AKD present in the sheet will be bound to cellulose, leaving the remaining portion unbound. It is not to say that the unbound does not contribute to sizing because it does.

**Size Reversion**

The problem of size reversion has begun to become a major issue for companies converting to alkaline papermaking. However, until recently little work has been published as to the causes. One technique used in the industry to alleviate reversion is to increase the addition level of AKD. Although this does not solve the problem it helps in keeping the paper in specification at the reel. It has been documented that some wet-end additives will interfere with sizing response. These include surface-active agents such as defoamers, dispersants, and biocides (28). Moyers (29) found that the interaction of precipitated calcium carbonate (PCC), size-press starch, and contaminants can have a devastating effect on AKD sizing response. Other papermaking variables including storage conditions can also promote size reversion. Studies found that high alkalinity in the papermaking system will promote reversion. Little has been done to compare the relationship between light exposure and sizing.

As mentioned earlier, there are no proven theories to the cause of size reversion. There are several beliefs on the mechanism, including the following ideas. Novak and Rende (28) attributed the loss in sizing due to cleavage of the ester bond to the cellulose. Patton (30) believed that the instability of AKD’s long chain alkyl groups in the presence of a moist environment cause them to reorganize in a manner that reduces the size response.
PROBLEM STATEMENT

Several factors have been studied including filler type and amount, defoamers, retention aids, biocides, and process conditions. The effect of light exposure has not been studied and documented to any degree. It is this author's belief that light degrades the bound and unbound portions of AKD which leads to size reversion. The primary objective of this study will be to prove this statement.

EXPERIMENTAL DESIGN

The first stage of this experiment involved examination of the chemical structure of AKD. This was done by forming a thin film of the sample and exposing a portion to direct UV light. The samples were then analyzed using infrared spectroscopy. The second stage of the experiment involved AKD addition to handsheets. The samples were then exposed to indoor light and UV light using the fade-ometer. Throughout the experiment paper testing of the samples included Hercules Size Test to determine the sizing response. The brightness and L, a, b color was also analyzed to determine if there is any change in the color due to the formation of color bodies.

Infrared Spectroscopy

The first stage of the experiment involved looking at the structure of the size after exposure to UV light. A thin film of the AKD was formed on a glass plate. A portion of the sample was kept in dark storage and the rest of the sample was exposed to UV light in the fade-ometer. Samples were exposed for 30, 60, and 120 minutes. For analysis of structure, the IR photo acoustic method was employed. Assistance in operating the instrument was gained through Dr. McKarville.

Papermaking and Size Addition

The papermaking furnish consisted of a blend of 70% hardwood and 30% softwood. No filler, retention aids, biocides, or any other typical chemical additive were used. The simplicity of the furnish allowed for the investigation of only the effect of light
on sizing response and not any of the other variables. The AKD sizing agent used was Hercon 75 which is a slightly cationic, self promoting dimer. This was added to the proportionator tank of the handsheet mold and allowed to mix for ten minutes before making handsheets. Low level sizing consisted of 0.15% and 0.20% active ingredient based on dry fiber. Intermediate level of addition was 0.25%, with the highest levels being 0.38% and 0.50%. The 0.25% was also repeated to check the reproducibility of the results. The specification on the sheet was 3.5 +/- 0.2 gram sheets. This tight tolerance was maintained to limit the effect of basis weight fluctuations on sizing level. The sheets were wet pressed once and dried two times through the drier can. Temperature of the drier can was kept at 250 °F to ensure curing, spreading, and anchoring of the AKD.

**Light Exposure Conditions**

Two different light exposures were employed. The first involved exposure to indoor laboratory lighting. Samples were placed felt side up on counter surfaces approximately five feet from the light source. The room was kept lit for a total period of fifteen weeks. The second lighting exposure utilized the laboratory fade-ometer for accelerated light aging. One hour of exposure is roughly equal to 24 hours of direct sunlight (31). The fade-ometer was run at the same operating conditions that were used for size structure analysis. Line voltage impressed was 192 volts at 80 degrees Fahrenheit. Samples were then allowed to age and then equilibrate under humidity controlled conditions before paper testing.

**Paper Sampling**

Samples from the indoor lighting experiment were tested at one week intervals for fifteen weeks of exposure. Samples exposed in the fade-ometer were tested at one hour intervals for a total duration of ten hours.

**Paper Testing**

The chief test utilized to determine sizing response and ultimately reversion was the Hercules Size Test. Standard No. 2 ink (1% formic acid) was used with the tester set
at 80% reflectance. A brightness test was performed on all samples in accordance with Tappi Standard T-452.

**Materials**

Components necessary for performing this study included pulp samples, AKD sizing chemical, and laboratory equipment. The hardwood and softwood pulp necessary came from the wet lab inventory as dried sheets. The laboratory equipment used included the Valley Beater, Noble & Wood system, humidity controlled laboratory, Fade-ometer, Hercules Size Tester, and the infrared spectroscopy equipment. Permission from Dr. McKarville of Chemistry department has been attained for the use of the IR equipment.

**Statistical Analysis**

Statistically, the sample sets were compared by their standard deviations. The percent that the standard deviation represents of the mean has also been calculated. The major source of error in this experiment is maintaining a consistent size level between handsheets. Each sheet has been designated with a numerical ID and was used throughout the duration of the testing procedure. This minimizes the error between using random sheets during each testing period. The Hercules Size test also has a standard error associated with the instrument, and represents a portion of the standard deviation.

**PRESENTATION AND DISCUSSION OF RESULTS**

**IR Spectroscopy**

Results for IR spectroscopy are found in Figures 1-4 in the Appendix section of this report. Figure 1. illustrates the IR structure of AKD with no exposure to UV light. Figures 2-4 show the IR structure after 30, 60, and 120 minutes of exposure. The distinguishing bands of 1200 to 2000 cm\(^{-1}\) is representative of water, along with the bundled peaks from 3400 to 4000 cm\(^{-1}\). The close double peak at just below 3000 cm\(^{-1}\) is most likely an alkane (carbon - hydrogen bond). The tallest peaks around 1400 to 18000
cm$^{-1}$ is a carbon to carbon double bond. A close examination of the data shows no significant change in the chemical bonding structure at any exposure level.

**Sizing Response - Indoor Light Exposure**

The primary focus of this experiment is the sizing response upon irradiation with both indoor light and UV light. Figure 1 shows the response for the lowest sizing level of 0.15% AKD addition. We see that the sample kept under dark storage showed a slight increase in the initial sizing level. This could possibly be due to curing of the AKD. The general trend however is a stable sizing level around 300 seconds HST. The sample exposed to the indoor light showed a sharp decrease in sizing after only one week of exposure, and after two weeks the response was not measurable.

**Figure 1. 0.15% AKD Addition Level**

![Graph showing sizing response over time](image)

Similar trends can be seen when we examine the 0.25% addition level in Figure 2. We see that the sample exposed to the indoor light showed the same general trend. After one week of exposure we lost more than 95% of the initial sizing level. Some sizing still remains, but after two weeks the sizing is completely degraded to only 1 second HST. The results for the dark storage sample are slightly different however. We see that after one week of storage more than 50% of the sizing level is degraded. The values continue
to show a downward trend throughout the duration of testing. It is important to note that although the dark storage sample is exhibiting some reversion, it still retains a base sizing level that does not degrade.

**Figure 2. 0.25% AKD Addition Level**

As the addition level increases the trend in the response begins to change slightly. Figure 3 displays the results for the 0.38% addition level and we see that the dark storage sample maintains a significantly high initial sizing level throughout the duration. After the first two weeks of exposure we see that the sizing had not significantly changed, and the slight decrease could be explained by the inherent error in the instrument. The trend begins to become more clear as the exposure time increases to six weeks of exposure at which the response begins to show a dramatic loss in sizing.

**Figure 3. 0.38% AKD Addition Level**
This same trend can be seen as the addition level is raised to the highest level of 0.50% in Figure 4. Again we see that the dark storage sample shows no change in sizing response throughout the testing period. We also see that the sample exposed to indoor light also showed no change until the last week of exposure. At this period we see the sizing level drop by more than 90% of the initial value. It is unfortunate that testing was not performed between the last stages of exposure to establish a better trend, however there were not sufficient samples left to perform testing.

**Figure 4. 0.50% AKD Addition Level**

At the completion of this stage of the experiment an emphasis was placed on attempting to repeat the results for one of the addition levels. The 0.25% addition level was chosen because of statistical reasons, which will be discussed later. Figure 5 illustrates the results of this trial. Again we see the same trend of size reversion in the sample exposed to indoor light, and a stable response of the dark storage sample. We note that there were some differences between this run and the original. The initial sizing level was three times higher than the original run. We also see that the initial size reversion as seen before was not as pronounced. Originally more than 90% of the sizing was lost within the first week, however this run did not drop to that level until after six
weeks of exposure. Although there were some slight differences, the overall trend was the same showing that the results could be reproduced.

**Figure 5. Repeat of 0.25% AKD Addition Level**

![Graph showing the effect of light exposure on sizing response.](image)

Finally, a comparison can be made by looking at all sizing levels together as seen in Figure 6. We see that the lower sizing levels, 0.15% and 0.25%, are the most dramatically effected by showing more than 95% size reversion within one week of exposure. The higher sizing levels show reversion, but not until the infinity testing of 13 to 15 weeks. We also see that the 0.25% repeat run had a much higher initial sizing level and took a longer time to exhibit reversion.

**Figure 6. The Effect of Indoor Light Exposure on Sizing Response**

![Graph showing the effect of light exposure on sizing response for different levels.](image)
**Sizing Response - Fadometer Exposure**

A second light source, direct UV light from the fadometer, was also used in order to establish a trend of size response. Figure 7 illustrates the sizing response for the three lower sizing levels of 0.15%, 0.20%, and 0.25% AKD addition. Again we see that the general trend is size reversion. The reversion for the lower sizing level is most pronounced, with the 0.25% taking the full four hours of exposure to reach 65% size reversion.

![Figure 7. Fadometer Results](image)

Figure 8 shows the same plot of the above results with the addition of the two higher sizing levels of 0.38% and 0.50%. We see that these higher levels were not effected after the scheduled four hours of exposure. The testing was then carried out to ten hours of exposure and the the samples still did not show any drop off in sizing response.

![Figure 8. Fadometer Results](image)
Another way to represent these results is by plotting them as a percent change from the initial sizing level. This is done for the 0.15% and the 0.25% addition levels in Figure 9. We see that the lower sizing level is showing a greater percent change from the initial sizing level than is the 0.25% level. This is further seen by examining the results for 0.38% and 0.50% where we see that there was a zero percent change in the sizing response.

![Figure 9. Fadometer Results - Percent Change](image)

**Brightness Results**

The brightness and color were examined as a side note to this experiment and samples from the fade-ometer were used for comparison of the brightness changes. In Figure 10 we see that the 0.15% level shows a slight decrease of one point in brightness. The 0.20% also shows a slight decrease of about a point in brightness. The 0.25% shows a change in the results by first decreasing a point in brightness after one hour of exposure, then a slight increase back to the same initial level during the last hour of exposure.
This same trend of showing a decrease during the first hour of exposure, then rebounding to a level above the initial sizing level can also be seen in the higher sizing levels. Figure 11 shows the results for the 0.38% and 0.50%. We see that the 0.38% actually has a net increase of one point in brightness. The 0.50% also shows a increase in brightness of about a half a point.
Statistical Analysis of Data

One of the objectives of this experiment was to determine if the results achieved were statistically comparable. To compare this data the standard deviation was calculated and compared between addition levels. Figure 12 shows the results for the lower sizing levels where the standard deviation was the highest. We see that with the 0.15% level the standard deviation was 290 seconds HST. More alarming is that this standard deviation represented more than 100% of the mean. This is saying that the standard deviation at this sizing level was greater than the mean. An even greater standard deviation is seen for the 0.25% addition level. In this case the standard deviation is 374 seconds HST, which represented almost 65% of the mean. The 0.38% showed a more acceptable result of a standard deviation under 100 seconds HST and representing less than 5% of the mean. A standard deviation of less than 100 seconds could possibly be attributed to the standard error of the instrument.

Figure 12. Statistical Comparisons between Addition Levels

![Graph showing statistical comparisons between addition levels]

The least predictable was the 0.25% addition level, and it was for the unusual high standard deviation that it was decided to repeat this run. Upon repeating this run we see that in Figure 13 the standard deviation was greatly decreased. In this graph we see that
at weeks one and four the standard deviation was the greatest, over 200 seconds HST. However it is still much improved from the initial run. A comparison of the initial standard deviation is seen in Figure 14. Here we see that the standard deviation was decreased by 75%. Although there were no changes in the procedure used, there was significant experienced gained in using AKD which helped in reducing the standard deviation originally seen.

**Figure 13. Statistical Analysis of 0.25% Reproduction**

![Graph showing statistical analysis of 0.25% reproduction](image)

**Figure 14. Comparison Between Reproducibility of Results**

![Graph showing comparison between reproducibility of results](image)
Summary of Results

The following summarizes the results seen in this experiment:

- IR spectroscopy showed no change in the chemical structure of AKD with two hours of UV irradiation.
- Size reversion was evident for all samples exposed to indoor light.
- The lower size levels showed the most dramatic size reversion. The higher levels were affected but to a lesser degree.
- Fade-ometer results duplicated the indoor conditions with the exception that the higher sizing levels were unchanged.
- A slight overall decrease in brightness is seen at the low sizing levels of 0.15% and 0.20%.
- High sizing levels of 0.25%, 0.38%, and 0.50% display a net increase in brightness values.
- Repeating the 0.25% addition of AKD confirmed that the results were reproducible.
- The repeat of 0.25% also indicated that the experience gained through working with AKD proved that the standard deviation could be decreased.
Conclusions

This experiment gives new light on the area of size reversion and opened ours eyes to a variable that had not been studied. Although this does not prove what causes size reversion, or more importantly give recommendations at how to eliminate the problem, it does help in our understanding of the situation. Even if the problem could not be eliminated, this experiment helps in establishing base line data for the trend of AKD and sizing response. The following statements sum up my beliefs on size reversion and how they relate to light exposure.

- IR spectroscopy results indicate that since the structure of AKD does not change upon irradiation that the mechanism of reversion involves the ester bond between cellulose and AKD.

- Size reversion in the presence of light is caused by the light acting as an energy source to form a hydrolysis of the unbound AKD. This belief is based on the fact that as much as 85% of the AKD can be unbound and that the hydrolysis product is a known desizing agent.

- The bound AKD is broken from the cellulose as the chain length of cellulose is decreased due to deterioration. Since cellulose chain bonds are being broken during light exposure it stands to reason that some of the bonds broken are the size to cellulose ester bond.

- In general, light speeds up the rate of the reverse reaction which is the breaking of the size to cellulose ester bond.
**Recommendation for Further Research**

Work is still needed in this area to specifically determine if this hypothesis stands in all cases. The following is a list of the areas related to this topic that could be further investigated.

1. The effect of light exposure on paper made from ASA and rosin.
2. The associated brightness loses exhibited in AKD, ASA, and rosin.
3. Prolonged irradiation in the fade-ometer.
4. The effect of different humidities and temperatures in the presence of light.

The following is a list of precautions to be followed in performing any further work in these areas.

1. Standardize all water used to 50 ppm hardness and 150 ppm alkalinity. These conditions are recommended by AKD suppliers and would help eliminate the variations in sizing levels between runs.
2. Control the conditions of the testing lab used for light exposure. This again would help eliminate the variables of temperature and humidity.
3. Perform testing on low sizing levels, preferably with an average HST below 500 seconds. High sizing levels beyond 2000 seconds are unpredictable and time consuming.
4. With the majority of reversion seen within the first week, testing should be performed at one day intervals for three weeks to establish a better trend. If higher sizing levels are used, such as 0.50%, testing should not start until after at least four weeks of exposure and extend out to 24 weeks to obtain a more accurate trend of the data.
BIBLIOGRAPHY


