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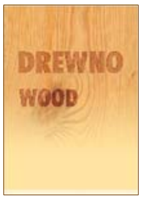

# Use of Glyoxal in Starch Based Sizing Mixture for Surface Modification of Paper

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Starches have been used as the surface sizing or coating material to enhance the strength and surface properties of paper. The main mechanisms behind their performance are physical and chemical interactions between cellulosic fibres and starches. In this study, the usage of glyoxal in an oxidized starch-based sizing mixture was studied to improve the chemical crosslinking due to the additional hydrogen and acetal bonds. The performance of glyoxal on starch sizing formula containing oxidized starch, styrene acrylate and polyaluminium chloride were studied in terms of water resistance and mechanical properties of paper. The results showed that the water absorption values ($Cobb_{60}$) decreased by 32% when adding glyoxal to paper coated with the reference solution, which contained only a styrene acrylate copolymer. Additionally, with this newly formulated surface sizing application, the tensile strength of the papers increased by about 16% compared with the reference.

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## Introduction

Cellulose, the main component of trees and plants, is the most abundant polysaccharide in nature. Paper and paperboard are renewable and recyclable materials composed of mainly cellulosic fibers and other constituents. Due to their recyclability, printing features, renewability, and mechanical flexibility, paper and related products are highly preferred, especially for different applications such as, packaging, printing and others.

Additionally, these materials are very hydrophilic inherently owing to hydroxyl groups, which contribute to the strength properties of paper via hydrogen bonds. Therefore, in addition to strength properties, water resistance is also required for packaging paper and boxes. To provide these properties, different sizing additives are used together with starch-based adhesives. Natural or modified starches used for reinforcing the paper are applied with before formation of paper sheet or with surface sizing at size press. The application of hydrophobizing agents and starch for the surface sizing of paper provides not only hydrophobicity but also surface strength due to the polymeric nature of starch and sizing agents. Sizing is generally limited to a surface region of paper that acts as a protective layer, which prevents water from entering the hydrophilic interior of the sheet [Bung 2004]. Surface sizing or hydrophobing agents can be divided conveniently into the following categories: rosin-

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based products, alkyl ketene dimer (AKD), synthetic polymeric materials and others. Of these, synthetic polymeric materials are the most important in modern surface sizing. Among of them, synthetic cationic styrene acrylate copolymers are popular, often used with alum or polyaluminium chloride. On the other hand, another important sizing agent, AKD, is applied to the surface; its use as a surface sizing agent is limited as a consequence of its waxy structure. Its waxy structure, which provides hydrophobicity, reduces the coefficient of friction of the paper surface, which can cause undesirable 'sliding' problems on the paper surface [Anderson 1997].

The most commonly preferred sizing materials are resin, starch, chitosan, polyvinyl alcohol, carboxy methyl cellulose and styrene acrylate emulsions [Özdemir et al., 2017].

Starch-based adhesives are preferred because of their biodegradability, abundance, and price. Raw starch can be modified chemically, physically, biologically or by using a combination of these methods. In different studies, latex binders or crude starch were modified with cross-linking agents such as glyoxal or different modification agents [Reddy and Yang 2010]. Glyoxal is the smallest dialdehyde, which has the ability to crosslink a wide range of polymers, including cellulose, PVA and starch [Zhang et al., 2010; Gadhave et al., 2019].

Bifunctional aldehydes such as glyoxal, glutaraldehyde and succinaldehyde were applied to the fabric surface as finishing agents for wrinkle resistance in textiles. Metal salts were also used as catalysts in the reaction between dialdehydes and cellulose. In addition, glyoxylated polyacrylamide (GPAM), which is obtained by reacting polyacrylamide with glyoxal, has been used in the paper industry as a temporary wet strength and dry strength agent [Lindström et al, 2005]. Glyoxal and glutaraldehyde solutions were used by Xu et al. in a specific molarity for the impregnation of kraft paper. It was reported that use of glyoxal increased the wet strength of paper by cross-linking between the cellulose molecule and glyoxal [Xu et al. 2001]. Glyoxal forms unstable hemiacetal bonds between cellulose molecules by bonding with the hydroxyl groups of the cellulose molecule. After the drying process, unstable hemiacetal bonds became stable and then crosslinking was completed [Kim et al. 1976]. If a catalyst is not used, the hemiacetal bond between cellulose and glyoxal provides paper temporary wet strength because of the water sensitivity of acetal groups. It was also found that glyoxal increased the strength of paper more than an glutaraldehyde addition [Xu et al. 2001].

Glyoxal and ammonium zirconium carbonate have been applied in the production of starch films as a crosslinking agent and it was stated that the produced starch films exhibited a more homogenous surface, higher thermal stability, mechanical strength and hydrophobicity. [Ni et al. 2019]. Polyvinyl alcohol, starch, carvacrol, and glyoxal have been used for starch films. An increase in the glyoxal concentration raised the water resistance and mechanical strength of starch films [Cheng et al. 2021]. The gelatinization process of sago starch was studied with the addition of different crosslinking agents: malonic acid, glyoxal, dicyandiamide, borax and polyethylene glycol [Mohamed et al. 2017].

In the open literature search, no study was found in which glyoxal and a styrene acrylate copolymer emulsion were used together with oxidized corn starch on the paper surface for coating or sizing purposes. In this study, a starch-based mixture was prepared by using oxidized corn starch, a styrene acrylate copolymer and glyoxal in different proportions and applied to the paper surface as a sizing mixture to change the strength and surface properties of paper.

## Material and methods

### Materials

In the study, oxidized corn starch was obtained from the TAT Starch company with the trade name Levosize 250. A hydrophobic polymer with the trade name Perglutin K650 (PERG), which is a styrene acrylate copolymer, was purchased from Kurita. Polyaluminium chloride (PAC) containing 17% $Al_2O_3$ and glyoxal (GLY) (~40% content water) were purchased from Akkim and BASF, respectively. The base papers used for surface treatment were fluting paper, with 100% recycled papers without any surface treatment, produced by Ankutsan, Inc.

### Methods

### Surface sizing

Oxidized starch was prepared in a beaker at a 9.5% (w/w) concentration. Then, heating and mixing processes were performed. The heating process was continued to reach 85 °C and the mixing process was continued for 5 minutes at this temperature. When gelatinization was observed, the solution was cooled to 70 °C and then styrene acrylate copolymer, polyaluminium chloride and glyoxal were added at different rates. After the starch solution is cooled to 70 °C, the pH value is about 7. When PAC, the styrene acrylate copolymer and glyoxal were added to the starch solution, the pH value of the starch solution was measured as nearly 5. Styrene acrylate emulsion products are often used with alum or polyaluminium chloride. Polyaluminium chloride (PAC) is as catalyst for styrene acrylate copolymer emulsion (PERG) by decreasing pH [Anderson 1997]. In a similar way, in this study PAC was used as a catalyst for the styrene acrylate copolymer by lowering the pH of the starch coating solution.

The prepared mixture was applied on preheated paper at 105 °C with a coating roller at 10 g/m$^2$. The base papers were preheated to have good impregnation of the sizing solution through the paper surfaces. After surface sizing, the papers were dried at 105 °C for 15 minutes. It is known that dialdehyde, glyoxal at 90 °C reacts with hydroxyl groups, forming an acetal bond between polyols and the medium required for this reaction to become acidic, which is inherently present in the solution [Cordes and Bull 1974]. Therefore, the drying temperature was chosen as 105 °C and after that the sized papers were cooled down to room temperature. The mixture formulas and constituents are presented in Table 1. A diagram of cellulose and starch crosslinking with glyoxal is given in Figure 1.

**Table 1.** Constituents of sizing mixtures

| Sizing formula codes | Constituents of sizing formulas |
|---|---|
| F0 | Base paper |
| F1 | 0.9% PERG + 1.3% PAC |
| F2 | 0.9% PERG + 1.3% PAC + 0.9% GLY |
| F3 | 0.9% PERG + 1.3% PAC + 1.7% GLY |

PERG: Perglutin K650, styrene acrylate copolymer
PAC: Polyaluminium chloride
GLY: Glyoxal

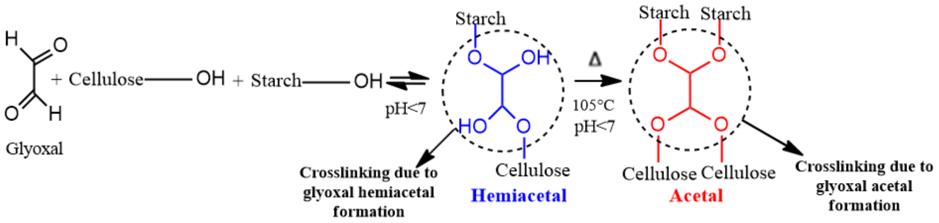


**Fig. 1.** Diagram of starch and cellulose crosslinking with glyoxal

## Paper tests

After sizing the paper surface, physical tests (Cobb 60) and mechanical strength tests (ring crush test and tensile strength test) were performed. The RCT test was carried out with a DEVOTRANS GPS 5 testing machine with a 48.6 mm disc according to the EN ISO 12192:2011 standard method. Tensile strength tests were conducted according to the EN ISO 1924-2:2008 standard method. The Cobb 60 value was determined according to the EN ISO 535:2014 standard method.

## Colorimetric analysis

The reference base paper and sized papers were analysed by means of an EXACT X-rite spectrophotometer. CIELAB was based on a three dimensional color universe that creates a Cartesian coordinate with L*, a* and b* axes. Three readings on the surface of the papers were taken using the CIE LAB L* (lightness), a* (redness), b* (yellowness) system in daylight conditions. ΔE represents the color difference measurement between the two colors in the color universe. The differences of the L*a* b* values were measured three times and average values were calculated by using Equation 1 below [Aydemir et al. 2021].

$$\Delta E^{*}_{ab} = \sqrt{(L^{*}_{2} - L^{*}_{1})^{2} + (a^{*}_{2} - a^{*}_{1})^{2} + (b^{*}_{2} - b^{*}_{1})^{2}} \qquad (1)$$

## Water contact angle analysis

The contact angles of distilled water on the paper surfaces were determined with a Goniometer (RAME-HART, Inc. Model no.100-00).

## SEM analysis

The surface of the papers were characterized by means of a scanning electron microscope (SEM; Zeiss; EVO LS 10), which was operated with an accelerating voltage of 5 kV with a detector distance of 13.5 mm and using magnification of 500 X.

## FT-IR analysis

The FT-IR spectrums of the coating formulas and starch were studied with a Bruker alpha II FT-IR spectrophotometer in the Quick Snap Platinum ATR module. Platinum ATR has a single reflection diamond ATR module. The spectrum was obtained in the range of the 4000 to 400 $cm^{-1}$ wavelength, at a resolution of 4 $cm^{-1}$.

## Results and discussion

## Mechanical and physical properties

To evaluate the performance of the sizing mixture, the ring crush test (RCT), tensile strength, and water resistance tests were carried out. The test results and the increase in grammage of the sized papers are shown below.

The reference sizing formulation (F1) with glyoxal affected the water resistance (COBB) of the papers in a positive way. The COBB results decreased from 65 to 45 and 44 g/m2, respectively, with an increasing concentration of glyoxal compared with the reference F1 (Figure 2.) Similarly, Lin et al. found that styrene acrylate and glyoxal usage in polyvinyl alcohol-unmodified starch films improved the moisture resistance of starch films [Lin et al. 2017]. Adding glyoxal to reference formula F1 containing styrene acrylate improved the water absorption resistance of the papers via the formation of acetal bonds. Since glyoxal reacts with hydroxyl groups of starch and cellulose at 105 °C, forming both inter-molecular chains and intra-molecular chains. The medium required for this reaction to occur is acidic, which is inherent in the solution. In a similar study, it was reported that is glyoxal reacts at 90 °C with the hydroxyl groups of polyvinyl alcohol and starch by forming acetal bonds in acidic media [Gadhave et al. 2019].

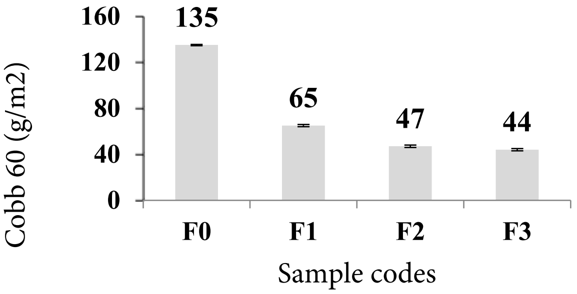


**Fig. 2.** Change in Cobb$_{60}$ values of base paper and sized papers

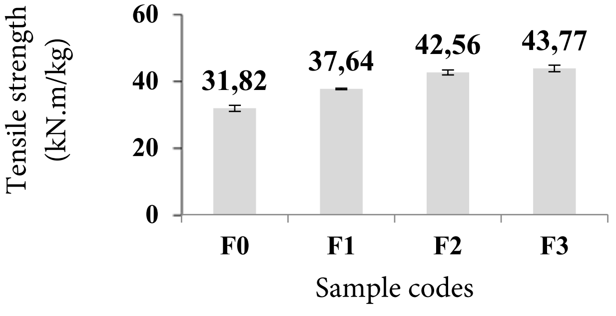


**Fig. 3.** Change in tensile strength of base paper and sized papers

The addition of glyoxal to the reference sizing mixture (F1) also affected the tensile strength properties of the paper. Increasing glyoxal in the formulation (F2 and F3) raised the tensile strength from 37.64 k.Nm/kg to 42.56 and 43.77 k.Nm/kg, respectively (Figure 3). Similar results were found in the literature. The impregnation of kraft papers with glyoxal improved the tensile strength because the hydroxyl groups of cellulose and starch react with glyoxal via the formation of acetal bonds [Xu et al. 2001; Gadhave et al. 2019]. When glyoxal is used with starch, acetal bonds between not only the starch hydroxyl groups but also cellulose and starch hydroxyl groups take place.

When the ring crush test (RCT) results were evaluated, it is clearly seen that adding glyoxal to the starch mixture increased the RCT values of the papers slightly from 1.20 kN/m to 1.34 kN/m and 1.32 kN/m, respectively (Figure 4).

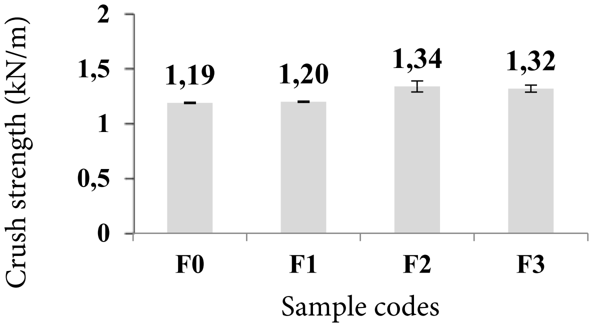


**Fig. 4.** Change in crush strength of base paper and sized papers

When improving paper strength properties, glyoxal forms unstable hemiacetal bonds between the cellulose molecules by bonding with the hydroxyl groups of the cellulose molecule. The unstable hemiacetal bonds became stable and then crosslinking was completed with heat treatment [Kim et al. 1976].

## Colorimetric analysis

The reference coating on the base paper and the addition of glyoxal to the reference coating were analysed by color difference using an EXACT X-rite spectrophotometer by means of ΔE values. The color differences are shown as in Table 3. It was observed that the sizing solution affected the base paper (F0) color by filling the pores of the paper. In additionally, increasing amount of the glyoxal in formulas affected the positive b* values that indicates the yellowness. The relationship between ΔE and the qualitative degree of color change are displayed in Table 4 [Hassan et al. 2021].

**Table 3.** Color change differences of paper surfaces

| **Sample codes** | **L*** | **a*** | **b*** | **ΔE** |
|---|---|---|---|---|
| F0 Base paper | 62.56 | 5.60 | 15.79 | 0 |
| F1 | 60.80 | 6.21 | 17.03 | 2.24 |
| F2 | 60.70 | 6.43 | 18.38 | 3.29 |
| F3 | 59.87 | 6.75 | 18.55 | 4.74 |

**Table 4.** Relationship between ΔE and qualitative degree of color change

| | |
|---|---|
| Very small difference | ΔE<0.5 |
| Fairly perceptible difference | ΔE<3 |
| Perceptible difference | ΔE<6 |
| Strong difference | ΔE<12 |
| Different colors | ΔE>12 |

## Water contact angle measurements of paper surfaces

The glyoxal addition to the reference sizing formula containing the styrene acrylate copolymer emulsion improved the hydrophobicity of the paper surfaces, and thus increased the water contact angles, as given in Table 5.

When low percentage of glyoxal adding in to the system, difference in water contact angles was detected as minimal. This situation of a minimal change depends on the hydrophilic nature of the starch and the addition of glyoxal to the starch sizing coating solution in low percentages comparing with the solid weight of the starch.

In the literature, the water contact angle of the film was measured as 69° for the starch films consisting of polyvinyl alcohol (PVA) and unmodified corn starch. The water contact angles of the papers were detected as 86° for the coated paper with the glyoxal and styrene acrylate copolymer mixture/PVA/starch (mixing ratio of 1:3:5) [Lin et al. 2017].When chitosan and glyoxal were used to increase the water contact angles of hand sheet papers by the impregnating process, the contact angle increased from 40° to 85° by coupling chitosan and glyoxal [Chen et al. 2013].

**Table 5.** Water contact angle measurements of sized paper surfaces

| Sample codes | Water contact angle (°) |
|---|---|
| F0 Base paper | 39 |
| F1 | 43 |
| F2 | 47 |
| F3 | 52 |

## FT-IR spectras of starch films

In this study, three sizing formulations were prepared by using oxidized starch with other chemicals. The F1 coating formula was chosen as the reference coating formula. The effect of glyoxal on the reference coating formula was investigated by means of FT-IR/ATR and the spectrums are shown in Figure 5. Since the quality of the spectra obtained by ATR-IR is highly dependent on the pressure applied to the sample [Vyorykka et al., 2006], an FTIR instrument with a diamond crystal single reflection ATR module was used in this study, thus allowing a constant amount of pressure to be applied to the sample. [Halttunen et al., 2001].

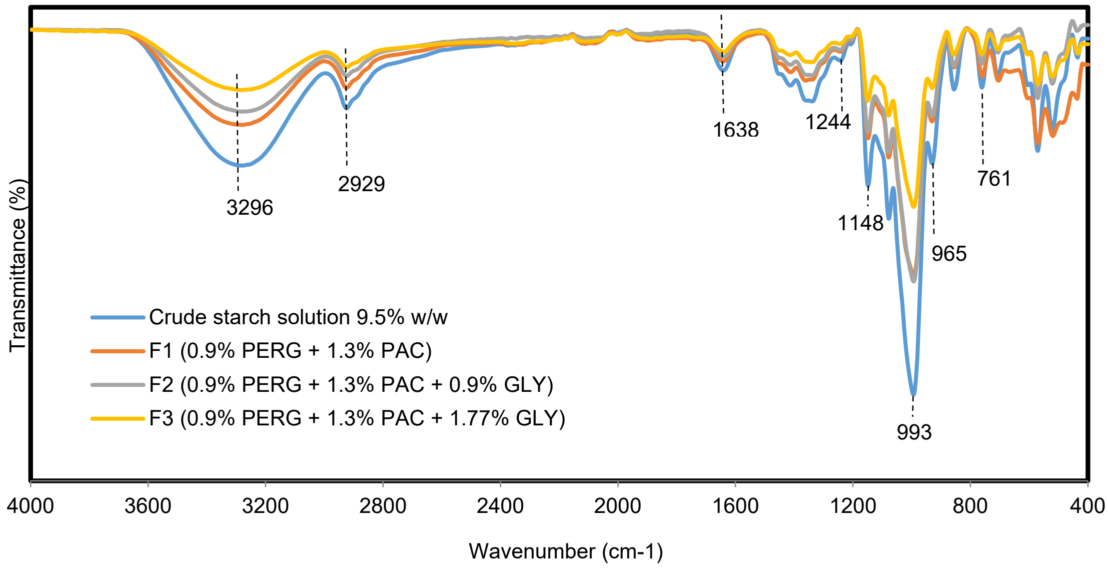


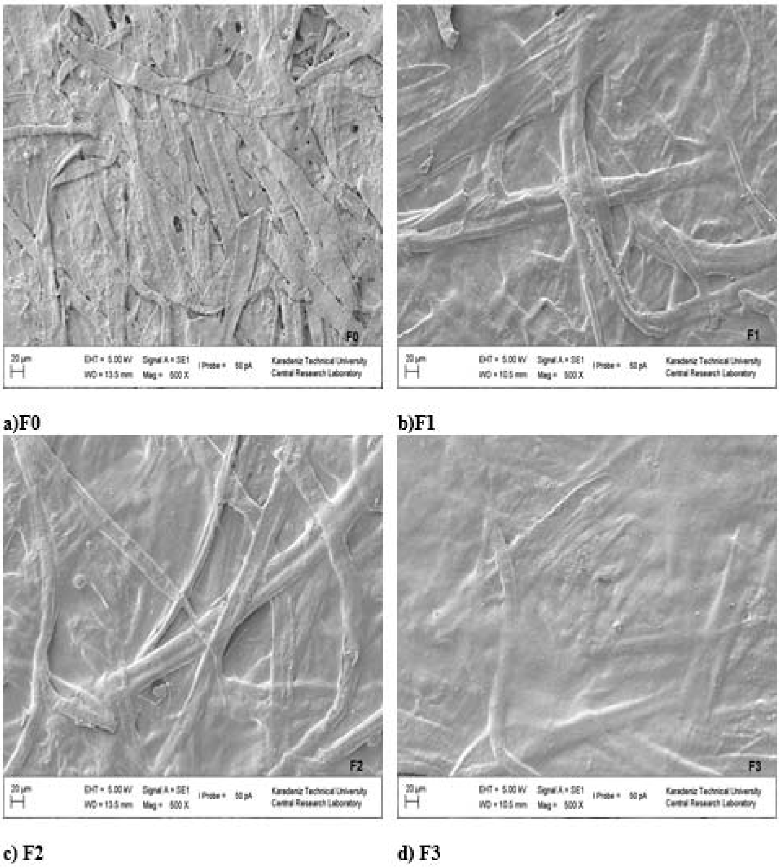
**Fig. 5.** FT-IR spectra of starch films

As seen in Figure 5, the FTIR spectra curves were similar because the elemental composition of the starch films dominated the other materials. The broad band of starch films at 3296 cm$^{-1}$ was due to OH stretching. [Detduangchan et al. 2014]. The absorption intensities of the broad bands were lower compared to the base starch solution film, indicating that crosslinking decreased the number of free hydroxyl groups in the films [Cheng et al. 2021 and Mittal et al 2020]. The peaks at 2929 cm$^{-1}$ refer to the C-H stretching vibration [Detduangchan et al. 2014]. The absorption band at 1638 cm$^{-1}$ corresponds to the intra-molecular hydrogen bond in oxidized maize starch [Hung et al. 2017]. By adding glyoxal, aldehydes undergo intermolecular dehydration reactions with free hydroxyl groups in starch, resulting in absorption peaks at 1244 cm$^{-1}$ and 1148 cm$^{-1}$, which were assigned to the C-O-C stretching vibrations [Cheng et al. 2021] and about 993 cm$^{-1}$ in the films, which was attributed to C-O stretching [Detduangchan et al. 2014]. The peaks at 761 cm$^{-1}$ and 965 cm$^{-1}$ were assigned to the vibrations of glucose pyranose units and the C-O vibrational stretching of the glucose unit, respectively [İbrahim et al. 2019].

## SEM analysis of paper surfaces

In the SEM micrographs of the papers given in Figure 6, it is observed that the sizing process was successful and the sizing mixture closes the gaps on the base paper surface by forming a continuous film. In this study, the same amount of sizing solution was applied on papers by roller at 10 g/m$^2$ for each sizing mixture. The untreated base paper had clear pores. These holes disappeared when the starch and sizing solution containing glyoxal was used. Based on the SEM micrographs, the surface structures of the papers sized with the reference starch solution (Figure 6b-F1) and 0.9% glyoxal-treated paper (Figure 6b-F2) were not so different comparing with the each other. However, a morphological difference became evident when 1.7% glyoxal-treated paper was compared against the base paper and other sized papers (Figure 6d-F3). When glyoxal was added to the reference sizing formula, the surface of the fibres was completely covered by a layer of sizing mixture to fill the pores of the base paper. Such effects were more dominant in the high glyoxal loaded paper (Figure 6d).

The SEM micrographs of the glyoxal-treated paper are clearly in correlation with the Cobb 60 and water contact angle measurements [Lin et al. 2019] and are proof of the glyoxal crosslinking with starch or fibres.



a)F0 b)F1

c) F2 d) F3

**Fig. 6.** SEM micrographs of base paper and sized papers, 500 X magnification, scale bar: 20 µm

## Conclusions

A glyoxal addition in to the surface sizing mixture containing starch, a styrene acrylate copolymer and polyaluminium chloride changed the water absorption values of paper in a positive way by rendering it water resistant. The decreasing Cobb 60 values and the increasing water contact angles are attributed to the crosslinking ability of glyoxal between the starch/starch, starch/cellulose or cellulose/cellulose interfaces. Another purpose of this study was to improve the water resistance of the papers by protecting the mechanical strengths of the papers. The results of the studies showed that the mechanical strength of papers such as, the ring crush strength and tensile strength grew with the application of glyoxal. Additionally, the color change of the paper surfaces were analysed and the results revealed that the added glyoxal affected the color of the papers. The FTIR spectrums of the films confirmed the occurrence of a crosslinking reaction between starch and glyoxal. The SEM micrographs of the glyoxal-treated papers clearly correlate with the Cobb 60 and water contact angle measurements.

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## List of standards

**EN/ISO 1924-2:2008** Paper and board - Determination of tensile properties - Part 2: Constant rate of elongation method (20 mm/min)

**EN/ISO 535:2014** Paper and board — Determination of water absorptiveness — Cobb method

**EN/ISO 12192:2011** Paper and board — Determination of compressive strength — Ring crush method