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Production of test lines paper with occ modified fibers and sodium caseinate crosslinker

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ABSTRACT

Currently, there is significant competition in recycling waste paper and using it for producing various types of paper used in the packaging sector. The paper derived from recycled pulps lacks good strength due to the gradual shortening of fibers and the reduction in bonding characteristics. Considering this challenge, in this study, after converting old corrugated containers (OCC) into pulp and then refining them, they were oxidized in an alkaline environment using hydrogen peroxide at varying percentages of 0, 0.5, 1, 2, 3, and 4 to create diversity and increase reactive groups. Sodium caseinate was then added to the pulp suspension at levels of 0.5, 1, 2, 3, 4, 5, 6, 7, 8, and 9 percent as a cross-linking agent. Finally, standard handmade papers were produced from them. The status of the formed functional groups was measured by FTIR spectrum, and the mechanical strength and water absorption of the produced papers were measured according to the standards of the Technical Association of the Pulp and Paper Industry (TAPPI). FTIR results showed that the oxidized pulp samples had high absorption at the wavenumber 1650, which corresponds to the carboxyl group. Additionally, the calculation of the carboxyl groups using the methylene blue absorption method indicated that the highest amount of carboxyl groups was in the 1 percent hydrogen peroxide treatment. The addition of sodium caseinate to the pulp suspension significantly increased the mechanical strength of the resulting papers.

1- Introduction

he reduction in mechanical strength of recycled paper due to the hornification of recycled fibers is a fundamental issue that must be addressed in the waste paper recycling industry. Compensating for the reduction in recycled fiber strength sustainably is essential and challenging [1]. Currently, there is significant competition for recycling waste paper and using it to produce various types of packaging paper. In some advanced countries, the recycling rate of paper has reached around 70 percent [2]. Recycled fibers hold a valuable position in paper and cardboard production [3]. One of the most important strategies to prevent deforestation and the shortage of wood fibers is to use non-wood fiber sources and waste paper [4]. From past to present and in the future, waste paper will account for a substantial portion of waste and byproducts from industrial and non-industrial processes, with recycling reducing the volume of waste. The recycling process for paper is highly important as it returns a useless material to the production cycle. Additionally, paper recycling can reduce the demand for virgin fibers globally, thereby decreasing the need to exploit valuable resources like forests. Old corrugated containers (OCC), used in the packaging sector, are among the fibrous materials accessible as waste paper. The consumption and production of this type of paper are increasing rapidly due to its suitable flexibility for various applications such as making food transport boxes [5].

Each part of the old corrugated containers (OCC) contains fibers that are physically and chemically different. Therefore, it is expected that the properties of the mix of these two types of fibers in recycled pulps will differ significantly from the properties of each type individually. Recycled fibers differ from virgin fibers. Test liner refers to the coated cardboard containing recycled fibers. Test liner coated cardboard is primarily produced in Central Europe and Asia. Various pulps are used to produce test liner coated cardboard. Because waste materials are used to produce this product, a multilayer structure is often preferred [6]. Another advantage of recycled paper is the reduced processing costs compared to virgin fibers. Processing recycled fibers consumes less energy than virgin fibers, and reduced energy consumption, in turn, leads to lower environmental pollution. Thus, it is evident that in the future, recycled fibers will play an increasingly important role as a valuable raw material for fiber-consuming industries such as papermaking, hygiene products, and cellulose derivatives. Recycled pulp may show less tendency to absorb water and have higher dimensional stability. Paper derived from recycled pulps lacks good strength due to changes in fiber structure caused by embrittlement, which reduces hydrogen bonds between fibers. The gradual shortening of fibers and reduced bonding properties limit the number of consecutive cycles for producing high-quality recycled paper [7].

Improving the performance of recycled paper can enhance its applications in various fields and contribute to resource savings [8]. Oxidation of fibers using hydrogen peroxide in an alkaline environment is one of the important methods of chemical modification. Studies in this area show that hydrogen peroxide can improve the tensile strength of fibers by up to 50 percent [9]. During the oxidation process, hydroxyl groups in cellulose can be oxidized to aldehyde or carboxyl groups to varying degrees [10]. Generally, oxidation improves the surface properties of cellulose fibers and is used in a wide range of applications such as papermaking, composites, textiles, and cellulose derivatives [11]. The surface charge of fibers plays an important role in their reaction with chemicals in an aqueous suspension. Surface modification of fibers using hydrogen peroxide can increase carboxyl groups and, consequently, the anionic charge of the fibers. Ionized carboxyl groups provide good absorption sites for resin molecules, and the higher the percentage of carboxyl groups in the fibers, the faster and more significant the absorption and retention of resins. The anionic property of the fibers makes them more likely to absorb cationic resins [12].

Researchers today are turning their attention to using natural resins to reduce the environmental damage caused by synthetic resins [13]. Different countries have recognized the importance of this issue and have begun using natural materials in their various industries [14]. Each year, about 18 percent of produced milk spoils due to insufficient storage and usage facilities [15]. Therefore, it is necessary to make more use of these materials to minimize waste. One potential use for milk is to produce casein and use it in various applications. Casein, a natural polymer, is extracted from the milk of mammals, especially cow's milk, and used in applications such as paint binders, paper sizing, wood coatings, additives in gypsum mortars, or as an adhesive [16]. Approximately 3 gallons of dilute milk are required to produce one pound of dry casein. Casein is separated and coagulated from milk by lowering the pH to about 2.6, its isoelectric point. Most often, casein is in powder form and must be mixed with water before use. In the chemical formula of casein, part of its structure consists of carboxylic groups, which carry a positive charge, and the other part consists of amine groups, which carry a negative charge. These two components neutralize the polymer's overall charge, which depends on factors such as pH. Casein is insoluble in water and alcohol. From the reaction of this substance with sodium hydroxide, sodium caseinate is obtained, which is soluble in water due to the presence of sodium ions, making it a more polar substance [17]. Increasing pH directly correlates with the total charge of sodium caseinate [18].

2- Materials and methods

2-1- Preparation of fibers from old cartons (occ):

OCC paper was obtained from waste paper collection centers. Contaminants such as adhesive tape and staples were removed. All layers of the carton were used to produce pulp. The cartons were cut into 5 square centimeter pieces and soaked in water for 24 hours, then dispersed in a disintegrator for 30 minutes. The resulting pulp was then dewatered on a screen with a 200-mesh sieve. After drying, the moisture content of the samples was measured, then packed in plastic bags and stored in a suitable environment until use. The moisture content of the pulp was determined based on the wet weight, according to the TAPPI standard T 412 om-94.

2-2- Chelation Process:

Hydrogen peroxide, as an oxidizing agent, can rapidly decompose and degrade when exposed to metal ions present in the pulp, leading to reduced efficiency. Therefore, after the oxidation stage with hydrogen peroxide, chelating agents such as DTPA[1] were used to remove heavy metals and prevent the decomposition of hydrogen peroxide. The chelating agent forms water-soluble complexes with metal ions, which are easily removed by washing, preventing the accumulation and reaction of metal ions with hydrogen peroxide. Thus, all pulps were chelated according to the conditions in Table 1 before bleaching.

Table 1. Pretreatment conditions for chelation

EDTA (%)	Temperature C°	time (minute)	Dryness percentage %	pH
0.5	60	30	60	5-5.5

2-3- Treatment with hydrogen peroxide

The treatment conditions with hydrogen peroxide are summarized in Table 2. Initially, the prepared pulp is placed in a polyethylene

1-diethylenetriaminepentaacetic acid

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bag. Then, sodium silicate and sodium hydroxide are dissolved in the required amount of distilled water and added to the pulp in the polyethylene bag, mixing thoroughly. Next, the specified amount of hydrogen peroxide is added, and the bag is sealed tightly. The bag is then placed in a warm water bath (Bain-marie) at 70°C for 90 minutes. The pulp is kneaded every 15 minutes to ensure uniform treatment. After the treatment, the pulp is washed with distilled water to remove the chemicals.

Table 2. Treatment conditions of occ pulp with different percentages of hydrogen peroxide

Dryness percentage (Percent)	Time (minute)	PH	NaoH/H_2O_2	Hydrogen peroxide (%)	Sodium silicate (%)	Temperature C°
10	90	10-11	0.8	0,0.5,1,2,3,4	3	70

2-4- Methylene Blue Adsorption Method:

To determine the amount of free carboxyl groups in oxidized pulps, the methylene blue adsorption test was employed. In this test, 0.5 grams of a pulp sample with a specified moisture content is suspended in 25 milliliters of methylene blue chloride solution and 25 milliliters of borate buffer at 20°C for 1 hour. The mixture is then filtered. Following this, 10 milliliters of 0.1 N HCl is added, and the volume is adjusted to 100 milliliters with water. The concentration of the methylene blue in the solution is then determined photometrically using a calibration curve at a wavelength of 664 nanometers. The total amount of free (unadsorbed) methylene blue is determined based on the laboratory results.

Formula 1:

$$\left(\frac{gr}{mmol}\right) \text{Carboxyl Groups} = \frac{7.5 - A * 0.00313}{E}$$

A: The total amount of free methylene blue(mmoL/ COOH)

E: Dry weight of dough sample (gr)

2-5- Treatment with Casein:

Sodium caseinate used in this study was obtained from Sigma-Aldrich and was added to the suspension of recycled OCC fibers oxidized with hydrogen peroxide at various percentages. Finally, handmade papers were produced from these fibers, and their properties were evaluated.

2-6- Production of Handmade Paper and Measurement of Its Properties:

To achieve the strength characteristics of the pulp and to measure its physical properties, as well as to evaluate the potential of the produced pulp for industrial processes, handmade paper was made. The 120-gram handmade papers were prepared according to TAPPI Standard 02-sp 205T. Given the basis weight of 2.4 grams per dry pulp, the amount of dry pulp required to produce the standard handmade paper was determined. The desired papers were produced using a laboratory hand papermaking machine.

The properties of the produced papers were measured according to the following standards:

- **Water Absorption Test**: Conducted according to TAPPI Standard 04om 441T using the Cobb method.
- **Tensile Strength**: Measured according to TAPPI Standard 01OM-494T.
- **Bursting Strength**: Evaluated according to TAPPI Standard 02OM-403T.
- **Tearing Strength**: Assessed according to TAPPI Standard 04OM-414T.
- **Air Permeability**: Measured using the Gurley method based on the volume of

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air passing through per unit time and according to TAPPI Standard T 460 om-96.

2-7- Statistical Analysis of Data:

For the statistical analysis of this study, the SPSS software was used. The data were analyzed as factorial experiments within a completely randomized design using analysis of variance (ANOVA). Mean comparisons were conducted using the Duncan test at a 95% significance level.

3- Discussion and results:

3-1- FTIR Spectroscopy:

Figure 1 shows the FTIR spectra of oxidized fibers at various levels and non-oxidized fibers. The comparison of peaks in the wavenumber range of 1650 cm^{-1}, which corresponds to carboxyl groups, indicates that carboxyl groups can be formed during the cellulose oxidation process with oxidizing agents [19]. Therefore, Figure 1, which illustrates the status of carboxyl groups before and after oxidation of fibers by different levels of hydrogen peroxide using FTIR spectroscopy, shows that the comparison of peaks in the wavenumber range of 1650 cm^{-1} reflects changes in the amount of carboxyl groups in the treated and control samples. Figure 2 presents the measured amounts of carboxyl groups in the treated and control samples. As shown, the highest amount of carboxyl groups is observed in the 1% hydrogen peroxide treatment, while the lowest amount is found in the control sample. Studies by Bhardwa and Nguyen (2005) indicate that increasing the percentage of hydrogen peroxide does not affect the formation of carboxyl groups [20], which is consistent with the results obtained in this research.

3-2- Analysis of Carboxyl Groups:

As shown in Figure 2, increasing the amount of hydrogen peroxide has led to a decrease in the amount of carboxyl groups. By comparing Figures 1 and 2, the changes in the carboxyl group content in the treated samples relative to the control sample can be confirmed. The carboxyl group is an ionizable functional group present in wood fibers, and during alkaline degradation, carboxylic acid groups are produced at the reducing ends of cellulose and hemicellulose chains. These groups can also be formed through oxidative treatments such as bleaching with hydrogen peroxide. Hemicelluloses naturally contain these carboxyl groups [21]. During the oxidation process, hydroxyl groups in glucosides and cellulose monomers can be oxidized to aldehyde or carboxyl groups. Therefore, one effective method for increasing the carboxyl group content in fibers is the bleaching process using hydrogen peroxide [10].

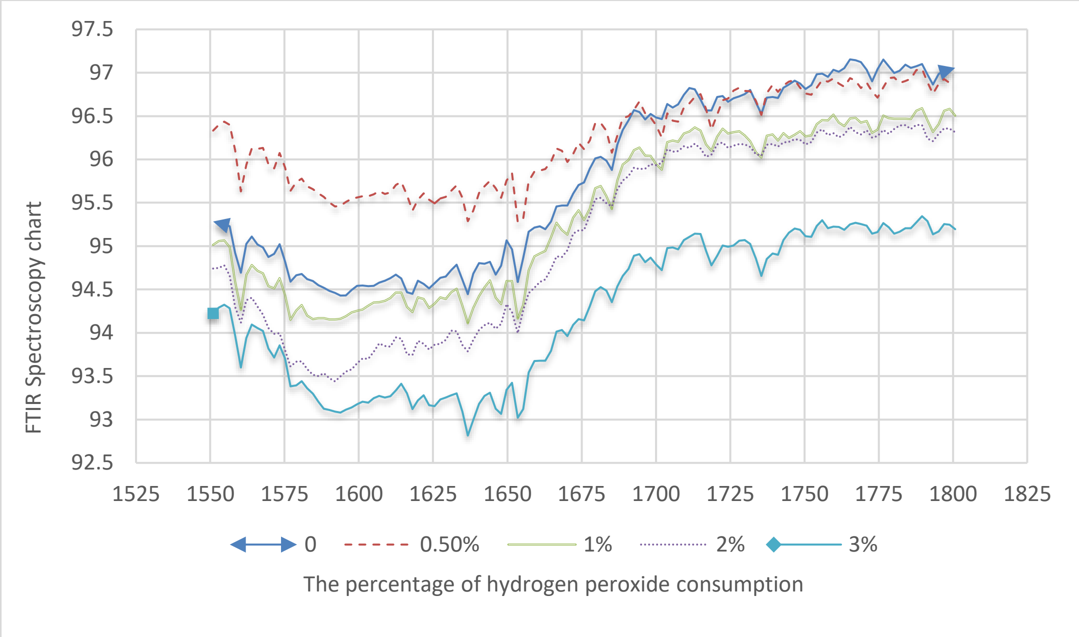

Figure 1: FTIR spectroscopy related to different levels of surface modification of occ fibers by hydrogen peroxide

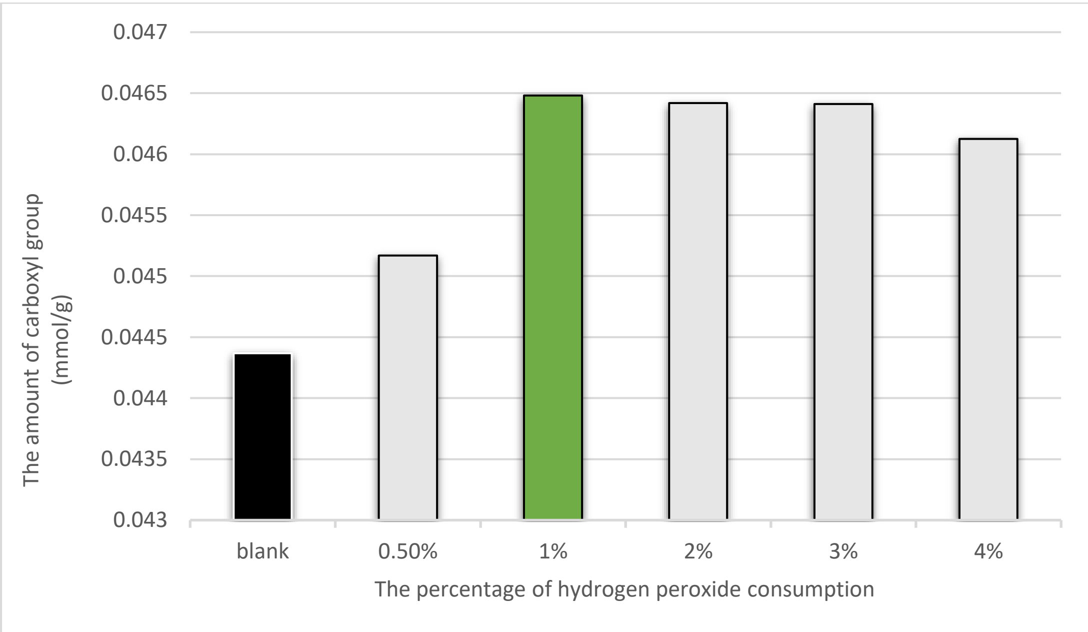

Figure 2: The amount of carboxyl group of rocc dough in different hydrogen peroxide treatments

3-3- Mechanical Properties:

The results of the impact of surface modification of fibers with hydrogen peroxide and the use of cross-linking agent sodium caseinate on the indices of tensile strength, burst resistance, and tear resistance show an increase in resistance compared to the control sample. Statistical analysis indicates that the differences are significant at a 95% confidence level (Figures 3, 4, 5, 6). Figure 3 illustrates the burst resistance index of handmade papers made from oxidized fibers with sodium caseinate. As shown in the figure, the use of sodium caseinate increases burst resistance in the papers, with the lowest resistance observed in the control sample and the highest at 3% sodium caseinate. However, with further increases in sodium caseinate consumption, a relative decrease in burst resistance is observed.

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Burst resistance testing is the oldest and most common method for measuring resistance in various types of paper. Factors affecting burst resistance include the type of fibers, the inherent strength of the fibers, the number of fibers per unit area of the paper, and the degree of bonding between the fibers[22]. Generally, burst resistance and tensile strength are closely related, and in both tests, the impact of fiber bonding and network density can be well observed[23]. An increase in hydrogen bonding and bonding surface area can enhance the fiber network strength and prevent fiber slippage[24. The modification of fiber surfaces with hydrogen peroxide can increase the carboxyl functional groups. Carboxyl groups are functional components containing a carboxylic acid group. When introduced into the paper matrix, they create additional sites for fiber interactions. Carboxyl groups, in addition to increasing hydrogen bonding between cellulose fibers, can also facilitate the formation of ester bonds. These bonds are strong forces that enhance the number and strength of connections between fibers, reinforcing the paper structure. Consequently, the paper becomes more resistant to tensile forces. The optimal increase in carboxyl groups significantly impacts the tensile strength of the paper. Enhanced fiber bonding strengthens it against mechanical stresses. The tensile strength index depends on many factors, which ultimately affect the number and quality of fiber connections. The greater these capabilities, the higher the tensile strength[25][26]. Internal fiber bonds, including hydrogen bonds between macromolecules of carbohydrate fibers, are key factors in determining paper tensile strength. The amount of hydrogen bonding is related to the accessibility of polysaccharides to each other and to surface functional groups such as carbonyl, hydroxyl, and carboxyl[27][28][29]. Increasing these can improve fiber network connections, making tensile strength a good indicator of fiber bonding. On the other hand, hydrogen peroxide may potentially disrupt fiber surfaces and increase surface porosity due to lignin removal and dissolution of other wood components like hemicelluloses, leading to increased fiber contact area and, consequently, enhanced fiber bonding and paper resistance properties such as tensile and burst resistance. Additionally, the alkaline environment in this process increases fiber swelling and surface area, which improves overall paper resistance[30]. The surface charge of fibers plays an important role in their interaction with chemicals added to the fiber suspension. Carboxyl groups, being ionizable functional groups present in wood fibers, have a significant impact on fiber surface charge. The amount of carboxyl groups in the paper pulp significantly affects resin performance. Anionic carboxyl groups provide favorable sites for the adsorption of cationic resin molecules, leading to faster and longer-lasting resin absorption with higher carboxyl group percentages in fibers. The anionic property of fibers increases their tendency to adsorb cationic additives. As shown in Figure 4, the use of sodium caseinate polymer as a cross-linking agent increases the tensile strength of the samples compared to the control. Casein is a milk protein with a complex and diverse structure. This protein consists of both surface and internal structures that interact with other molecules. Casein contains hydroxyl and carboxylic groups that bond with other molecules, including paper fibers. These bonds can be formed through hydrogen bonds, Van der Waals forces, and electrostatic forces, which help reinforce the paper structure and increase its resistance to tensile forces[31]. In papermaking, adding casein to the pulp suspension can affect the paper's structure and properties. Molecular interactions between casein's hydroxyl groups and cellulose's hydroxyl groups can strengthen hydrogen bonds and enhance paper stability and strength. These interactions between casein and cellulose can improve the internal structure of the paper and increase tensile resistance. Therefore, casein can act as an effective cross-linking agent in the paper structure, enhancing its strength and stability against various forces. Studies by Lalitha et al. (2020) confirm the presence of hydrogen bonds between casein and cellulose fibers, which aligns with the results of this study[31]. Thus, modifying paper fibers with hydrogen peroxide to increase carboxyl groups and the anionic charge of fibers, combined with the addition of casein with cationic charge in an alkaline environment, can

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create electrostatic bonds between fibers. Both hydrogen and electrostatic bonds play a role in strengthening the paper structure. Hydrogen bonds typically form between hydroxyl groups in cellulose and hydroxyl groups in casein[32][12]. These bonds create molecular connections between the two chemicals, reinforcing and strengthening the paper structure. Additionally, electrostatic bonds between positively charged (cationic) groups in casein and negatively charged (anionic) groups in the paper fibers can stabilize the structural integrity between fibers and enhance the indices of tensile and burst resistance.

Figures 3 and 4 show that increasing the amount of sodium caseinate in the paper results in a relative decrease in resistance. Excessive addition of casein may cause an imbalance in the paper structure. Changes in molecular structure or internal fiber surface equilibria with increased amounts of cationic material and changes in the overall charge of the fiber suspension may lead to degradation or reduced bonding between fibers, resulting in decreased paper resistance. Figure 5 shows the tear resistance index of handmade papers made with oxidized fibers and sodium caseinate polymer. The results indicate that the use of sodium caseinate in oxidized OCC recycled papers increases tear resistance, and the difference is statistically significant at the 5% level. Among the factors affecting tear resistance, fiber length and inherent fiber strength are the primary influences. However, if both factors are similar, fiber bonding can significantly affect tear resistance. Adding casein creates additional bonds between fibers, leading to a limited increase in tear resistance. Thus, increasing the amount of casein in the paper may result in a slight decrease in tear resistance, indicating minimal changes in the inherent fiber strength when using casein.

Measuring the permeability properties of paper is another important characteristic of paper products. Paper resistance to air permeability is a crucial component in assessing paper features. Measuring the rate of air passage through the paper can serve as a reliable indicator of relative porosity, softness, and roughness. Methods such as the Gurley method are used to measure the volume of air passing through within a specified time. An increased air permeability rate indicates a paper with higher porosity and permeability. Conversely, a reduced rate indicates higher resistance to air permeability and different overall paper characteristics[33]. Therefore, a reduction in air permeability suggests filling or blocking of paper pores, leading to increased resistance to air flow through the paper.

3-3- Barrier Properties:

Figure 6 illustrates the effect of different levels of sodium caseinate on the air permeability of paper treated with hydrogen peroxide. As shown, the relationship between sodium caseinate consumption and air resistance in the paper is direct. Increased sodium caseinate leads to decreased air permeability, indicating that the surface pores of the paper are engaged, and air passage is somewhat controlled. With enhanced fiber bonding and reduced porosity between fibers, the paper's resistance to air permeability increases. Strengthening the bonds between paper fibers improves the internal structure of the paper, which can reduce the void spaces between fibers. This reduction in voids decreases the likelihood of air passing through the paper. Moreover, casein molecules, by filling the gaps between fibers, act as a filler, especially at higher concentrations, leading to a more significant increase in air resistance.

The average values of water absorption for different treatments were statistically significant at the 95% confidence level. According to Duncan's test, the average values of water absorption are grouped into three categories. Additionally, Figure 7 shows the effect of different treatments on the water absorption of treated and control papers. As observed, all treatments increased water absorption in the handmade papers, with the control sample having the lowest water absorption.

Water absorption is a crucial method for evaluating paper quality and determining its ability to absorb and retain water. This test is used in various industries such as printing and packaging, food industries, and cardboard production. Moveydi et al. (2015) stated that fiber oxidation with hydrogen peroxide increases fiber hydrophilicity, which aligns with the results obtained in this study[12]. Carboxyl groups are known as hydrophobic (water-repellent) groups; however, increasing the number of carboxyl groups can alter the arrangement and formation of paper fibers. These changes might improve particle distribution in the paper structure and enhance the paper's ability to absorb and retain water. Changes in surface structure and chemical interactions with water can ultimately improve the paper's water absorption and retention capabilities.

Casein, compared to many synthetic polymers, has high polarity due to its hydrophilic nature, making it permeable to moisture[34][35]. Casein's structure, with hydroxyl (OH-) groups, exhibits hydrophilicity[36]. As a moisture stabilizer and structuring agent, casein enhances the paper's ability to absorb and retain water, and the results of this study support this finding.

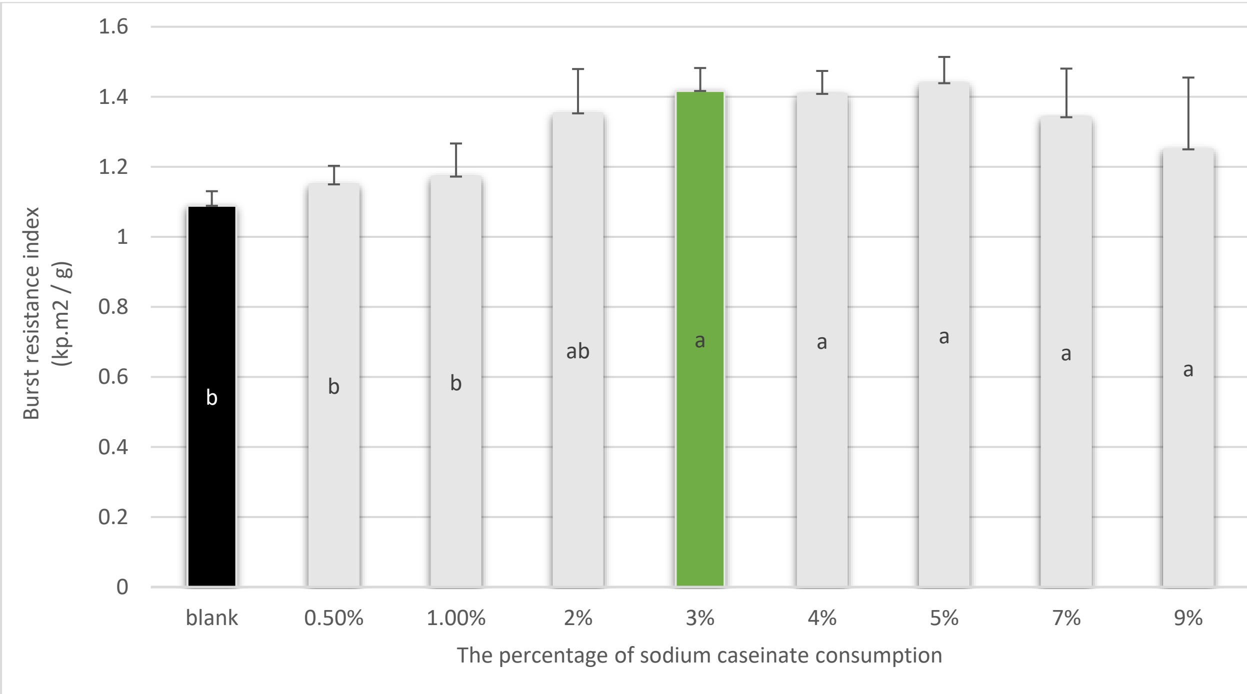

Figure 3: The effect of different levels of sodium caseinate on the bursting resistance index of handmade papers

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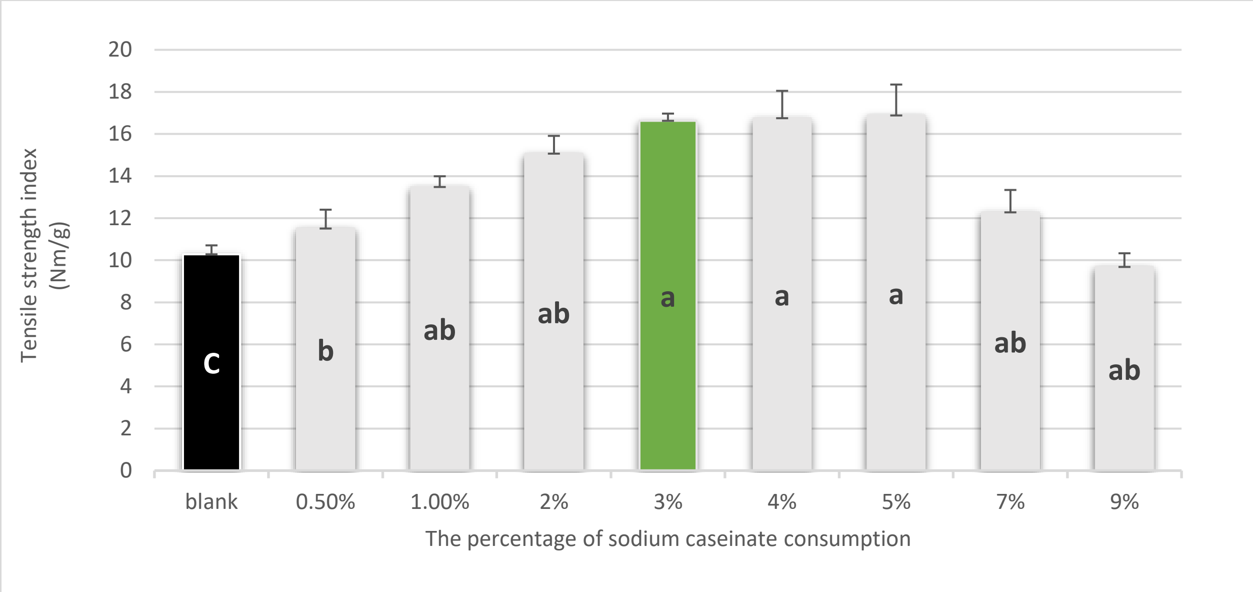

Figure 4: The effect of different levels of sodium caseinate on the tensile strength index of handmade papers

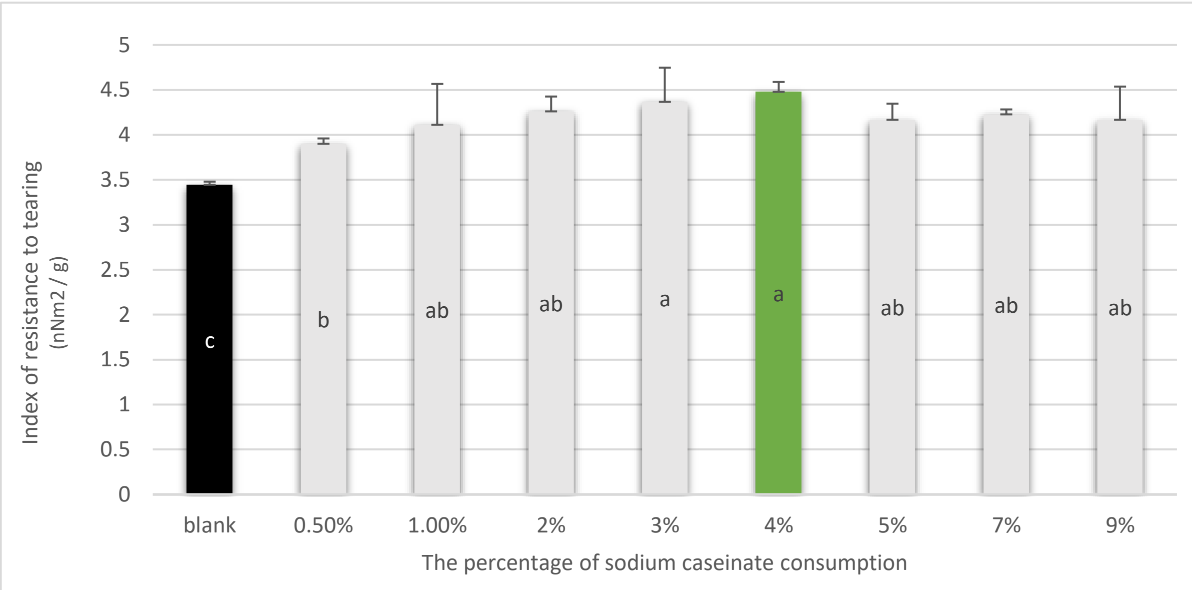

Figure 5: The effect of different levels of sodium caseinate on the tearing resistance index of handmade papers

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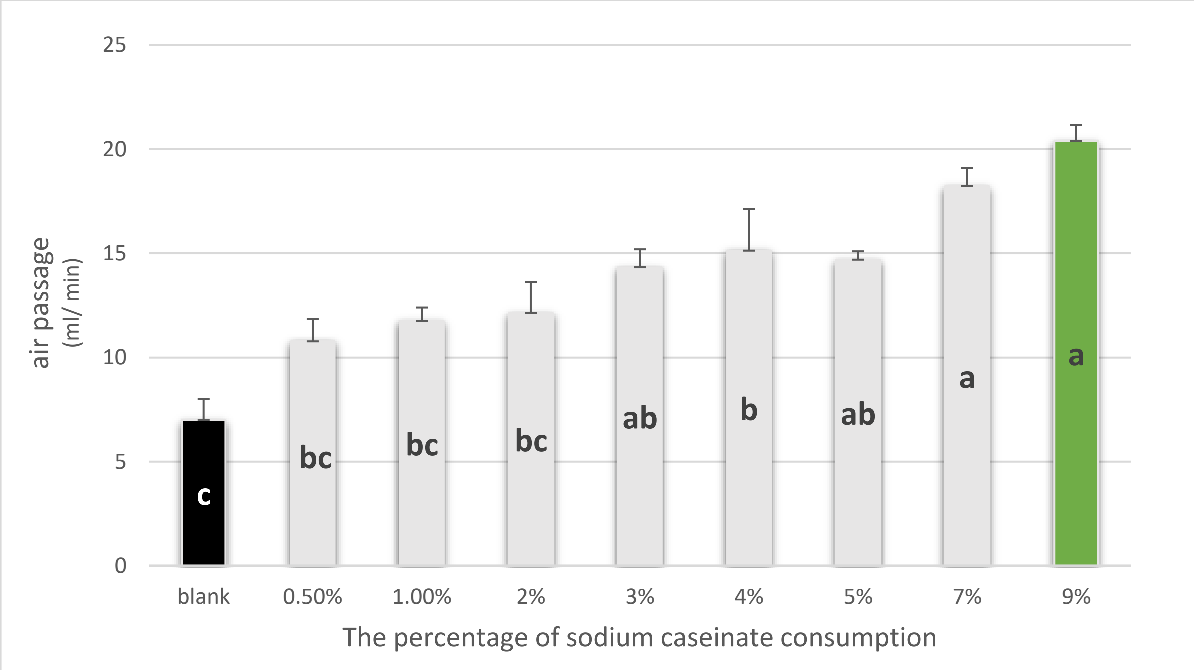

Figure 6: The effect of different levels of sodium caseinate on the air resistance index of handmade papers

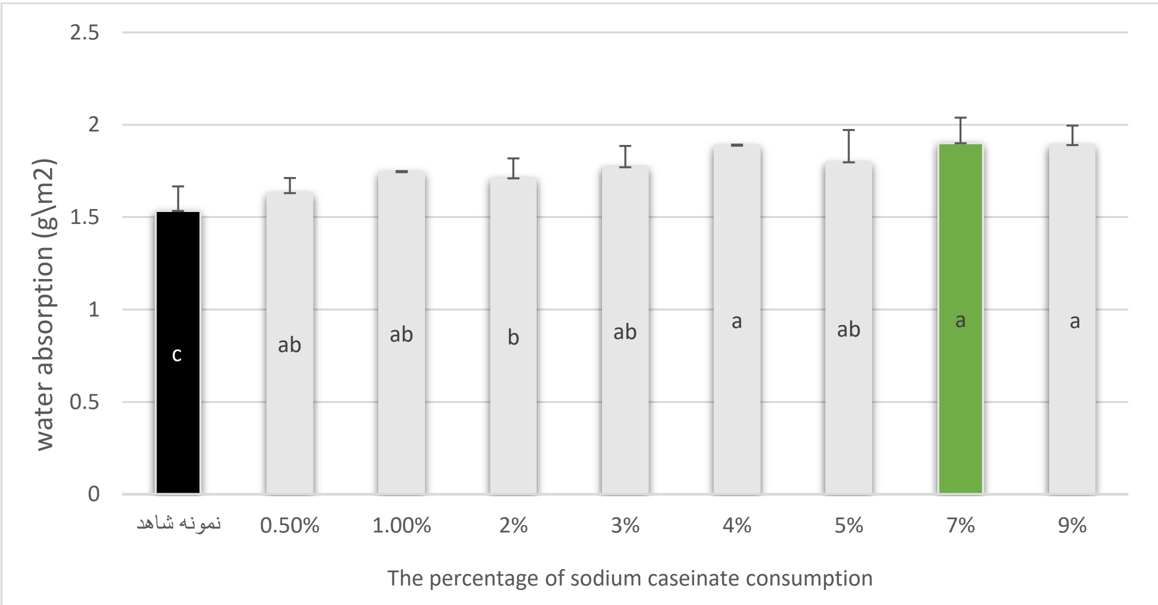

Figure 7: The effect of different levels of sodium caseinate on water absorption of handmade papers

4- Conclusion:

The results of this study demonstrate that the use of sodium caseinate as a cross-linking agent in the production of recycled paper can lead to significant improvements in both the mechanical properties and overall performance of the paper. While sodium caseinate is widely recognized for its applications in the food industry, this research highlights its potential as an effective cross-linking agent in paper production. It contributes to enhancing fiber bonding, thereby gradually improving the mechanical properties of the paper.

The increase in mechanical strength and stability of recycled paper enables its use in various industrial applications, particularly in packaging where high durability and strength

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are required. The application of hydrogen peroxide as an oxidative agent for pulp, combined with sodium caseinate as a cross-linking agent, results in enhanced characteristics of recycled paper.

Given the importance of environmental conservation and efficient resource utilization, the development of recycled papers with improved properties plays a crucial role in reducing natural resource consumption and supporting environmental sustainability. The findings of this study provide a basis for advancing new technologies in paper recycling and optimizing resource use, thus contributing to the development of higher-quality recycled paper products.

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مقاله علمی_پژوهشی

تولید کاغذ تست لاینز با الیاف اصلاح‌شده occ و اتصال‌دهنده عرضی سدیم‌کازئینات

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چکیده

در حال حاضر رقابت خیلی زیادی برای بازیافت کاغذهای باطله و استفاده از آن برای تولید انواع کاغذ مورداستفاده در بخش بسته‌بندی وجود دارد. کاغذ حاصل از خمیرهای بازیافتی بـه علت کوتـاه شـدن تدریجی الیاف و کاهش ویژگـی پیونـدیابی مقاومت خوبی نـدارد. با توجه به این چالش در این مطالعه پس از تبدیل کارتن‌های کنگره‌ای کهنه(occ) به خمیرکاغذ و سپس پالایش آن‌ها، جهت ایجاد تنوع و افزایش گروه‌های واکنش‌پذیر با درصدهای متفاوت ٤، ۳، ۲، ۱، ۰/۵، ۰ توسط پروکسید هیدروژن در محیط قلیایی اکسید شدند. سپس سدیم‌کازئینات در سطوح ۵،٦،۷،۸،۹ ،٤ ،۳ ،۲ ،۱ ،۰/۵درصد به‌عنوان اتصال‌دهنده عرضی به سوسپانسیون خمیر اضافه شد. درنهایت از آن‌ها کاغذهای دست‌ساز استاندارد تهیـه گردید. وضعیت گروه‌های عاملی تشکیل‌شده توسط طیف FTIR و مقاومت‌های مکانیکی جذب آب کاغذهای ساخته‌شده طبق استانداردهای انجمن فنی صنعت خمیر و کاغذ آمریکا (TAPPI) اندازه‌گیری شدند. نتایج FTIR نشان داد که نمونه‌های خمیر اکسیدشده جذب بالایی را در عدد موج ۱٦۵۰ داشته‌اند که مربوط به گروه کربوکسیل است. همچنین محاسبه مقدار گروه‌های کربوکسیل با روش جذب متیلن‌بلو نشان داد که بیشترین مقدار کربوکسیل در تیمار ۱درصد پروکسیدهیدروژن بوده است. با افزودن سدیم‌کازئینات به سوسپانسیون خمیر، مقاومت‌های مکانیکی کاغذهای حاصله به‌صورت معنی‌دار افزایش‌یافته است.