INVESTIGATION OF THE PROPERTIES OF COLD STORAGE BROWN CARDBOARD COATED WITH NANOPOLYURETHANE AND NANOCLAY

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This study aims to investigate the properties of cold storage Kraft liner brown cardboard coated with nanopolyurethane and nanoclay, comparing the effects of single-layer and double-layer coatings on its performance. The Kraft liner cardboard, produced by Pishgaman Company, was tested after being coated with a solution applied at a rate of 15 g/m². Coated samples were allowed to dry at room temperature for one day before being stored in a refrigerator at -15 °C temperature for periods of 2 and 4 months. A secondary coating was applied using a wire bar coater at a rate of 27 g/m² to enhance surface properties and further reduce water absorption. Results indicated that the coatings effectively penetrated the paper pores, significantly decreasing water absorption, particularly in double-layer coatings. Additionally, the thickness and smoothness of the surface increased, with notable improvements maintained even after freezing. While the tensile index, burst index, and crushing index of the liners showed a reduction, tear resistance increased, especially in the machine direction, with more significant changes observed in double-layer coatings. In conclusion, the use of nanopolyurethane and nanoclay coatings significantly enhanced the properties of Kraft liner cardboard, making it suitable for applications in food packaging, such as liquid packaging cardboard for juices and milk. Further studies are recommended to explore these coatings on various packaging materials over different time periods.

Keywords: cardboard, brown, cold storage, coating, nanopolyurethane, nanoclay

INTRODUCTION

Cold storage cardboards are suitable for the humid atmosphere of cold storage facilities, ensuring they remain effective without failure in such environments. The humid atmosphere of cold storage is ideal for preserving a variety of products over extended periods of time. Thus, the importance of using cardboard for cold storage applications cannot be overlooked. High strength is one of the essential properties of these types of cardboard, particularly for export-grade materials. When transporting products, such as fruits, vegetables, chicken, fish, meat and other protein items, the integrity of the packaging is crucial, especially given the varying conditions encountered during transportation. Export cardboard must possess a robust physical structure to protect the contents, ensuring they arrive at their destination safely. Additionally, the recyclability of cardboard enhances its appeal to environmentally conscious consumers.

The primary objective of food packaging is to maintain product quality from production to consumption. Quality loss often occurs due to the transfer of oxygen and water vapor, which can be mitigated by applying a coating barrier on the packaging.[1] Transitioning from synthetic to natural coatings presents an opportunity for recycling food packaging.[2] Although wax and polyethylene are commonly used in the packaging industry to impart desirable properties to packaging paper and containers, they are not biodegradable and pose environmental challenges.[3]

Typically, common coating barrier are made from expensive synthetic polymers like vinyl ethylene alcohol and polyvinylidene chloride. While these coatings are effective against moisture and oxygen penetration, their recyclability is limited. Current research is increasingly focused on developing biodegradable polymers for packaging applications.[4] The significant advantage of biodegradable coatings is their ability to separate easily from the surface through chemical or enzymatic processes, facilitating recycling.[5]

A pressing issue in the food packaging industry is the non-biodegradability of most packaging materials, contributing to municipal solid waste. Agricultural alternatives to polyolefin packaging are being developed, which can bolster the agricultural economy, while reducing reliance on petroleum products. Studies on paper coated with biopolymers have primarily focused on enhancing oil resistance, as many biopolymer films are hydrophilic and expected to perform well in low humidity conditions.[6] Substances of biological origin are biocompatible, biodegradable, and renewable.[7] A key benefit of using these substances is that increased absorption on fiber surface enhances fiber bonding and, ultimately, the strength of the resulting paper. However, common methods typically limit the absorption of these polymers.[8]

Rhim *et al*. found that coatings comprising biopolymers (such as alginate and soy protein) increased the thickness of paperboard from 9% to 16%, depending on the coating type and treatment method, resulting in a smoother and more homogeneous surface. They reported that the tensile index of uncoated paper decreased by 12.5% to 37.5% after coating, while the crush index remained unchanged. Moreover, the permeability and water absorption properties were also influenced by the biopolymer coatings.[9]

In the meat packaging industry, which includes chicken, fish, beef, and other protein products, multilayer cartons or compressed cardboard are commonly employed. Cold storage, often necessary for these products, introduces a humid environment, and the products themselves may contain moisture. Therefore, the cardboard coating used in meat packaging must be resistant to both product moisture and the conditions of cold storage. Unfortunately, currently available coated cardboards often lack the desired moisture resistance.

To address this issue, applying coatings to enhance moisture resistance and improve cardboard properties is essential. Natural renewable biopolymers, such as nanocellulose, nanographene, and various protein materials, such as zein, can effectively act as barriers to moisture and soluble substances, offering environmental benefits, such as recyclability, compared to synthetic petroleum-based polymers. Consequently, recent studies have focused on these materials in food packaging applications. This study, investigates the use of single-layer and double-layer coatings with biodegradable nanostructured compounds, aiming to evaluate the coating method for producing high-quality coated cardboard and exploring the differences between single-layer and double-layer coatings.

EXPERIMENTAL

Materials

In this study, Kraft liner brown paperboard was received from Pishgaman Company. The basis weight of this paper was 128 g/m². The nano-clay used included Clocite 30B (Southern Clay Products). The nano-polyurethane used was prepared by a knowledge-based company and its specifications are presented in Table 1.

Table 1
Specifications of nano-polyurethane

Appearance	Light yellow liquid
Type	Transverse self-bonding
Emulsion property	anionic
Total solids	33%
Viscosity	378 mPa.s
Particle size	10-70 nm

Base paper coating

First, the liners were placed on a layered board and coated with a spray gun at the rate of 15 g/m². The coated cardboard was restrained and dried at room temperature for one day to stabilize the coating on its surface. The samples were then placed in a refrigerator at -15 °C for 2 and 4 months before determining their physical and mechanical properties. To enhance the surface properties and further reduce the water absorption of the primary-coated liners, a secondary coating was applied using a wire bar coater, amounting to approximately 27 g/m². The coated samples were air-dried and subsequently stored in the refrigerator for 2 and 4 months. Finally, the samples were conditioned under climate conditions (27 °C and humidity 65% for 24 hours) according to ISO-187 for testing.

Figures 1 and 2 illustrate the process of restraining and positioning white-faced kraft papers for the application of the first coating using a spray gun, as well as their placement in the freezer. The composition of the coating formulation and other specifics are summarized in Table 3.

Figure 1: Method for holding and positioning Kraft liner brown paper for the application of the first coat using a spray gun

Figure 2: Placement of Kraft liner brown paper in the freezer for durations of 2 and 4 months

Table 2
Composition of secondary coating

Component	unit	value
Micro-kaolin	%	65
Ground calcium carbonate (GCC)	%	22
Latex (Styrene butadiene NS87 Simab)	%	10.5
Montmorillonite nanoclay	%	0.3
Dispersant (D200 mercury resin)	%	0.2
Carboxymethyl cellulose (CMC)	%	1
Ethylene glycol	%	1
Viscosity	mPa.s	700
Solids (S.C.)	%	64
pH		7.8

Table 3
Differently treated samples and their corresponding denotation

No.	Code	Description
1	b_0	Control brown liner
2	b_2	Control brown liner (freezing for 2 months)
3	b_4	Control brown liner (freezing for 4 months)
4	c_{10}	Single-coated brown liner
5	c_{12}	Single-coated brown liner (freezing for 2 months)
6	c_{14}	Single-coated brown liner (freezing for 4 months)
7	bc_{20}	Double-coated brown liner
8	bc_{22}	Double-coated brown liner (freezing for 2 months)
9	bc_{24}	Double-coated brown liner (freezing for 4 months)

Measurement of paper properties

Physical properties, including thickness (T411 om-89), water absorption (T441 om-96), and surface smoothness (T555 om-04); as well as mechanical properties, including tensile index CD and MD (T404 om-92), burst index (T403 om-02), tear index CD and MD (T404 om-04), crushing index CD and MD (T818 om-97) and brightness (T403 om-91), of the prepared papers were measured according to TAPPI standards. The contact angle was measured to assess the wettability of various paper sheets, using water as the probe liquid.[10] The wetting test was conducted using a CAM (Contact Angle Measuring) device developed at the Wetting and Fluids Laboratory of the Materials and Energy Research Center. Images were captured with a

DFK 23U618 USB 3.0 Color Industrial Camera, utilizing a 2X lens for enhanced detail.

Microscopic studies

Microscopic images were taken from the surface of control and coated samples at the Razi Metallurgical Research Institute, using field-emission microscopy (FEM) (XMU Mira 3), this being the newest and most advanced FE-SEM available in Iran.

Statistical calculations

The experimental design used in this study was completely random and SPSS 23 was used to process the results of the measurements. For data analysis, one-way analysis of variance was used and Duncan's test was used to compare the means.

RESULTS AND DISCUSSION

The analysis of variance was used to determine the statistical difference among the samples for each property. Table 4 shows the F-value obtained from this analysis and the significance level.

Paper thickness

One-way analysis of variance (ANOVA) revealed a statistically significant difference in the thickness values among the nine types of tested paper at a 5% probability level. The thickness values were categorized into four distinct groups across all treatments. As shown in Figure 3, the double-coated brown liner paper exhibited the highest thickness, while the control sample that was frozen for 2 months demonstrated the lowest thickness.

The thickness of the double-coated brown cardboard increased by 13.7%, compared to that of the control sample. The deposition of the aqueous coating solution on the cellulose substrate resulted in an increased thickness, indicating a loss of homogeneity and uniformity in the coating layer. This deposition led to the formation of a coating layer, with its thickness influenced by the nature of the polymer and the solid content in the coating solution. Notably, the addition of nano-sized components to the coating formulation significantly increased the thickness of the samples. Following the coating process, enhanced molecular contact between the coating compounds may weaken the compaction forces of the polymer chains, further opening the coating matrix and contributing to the increased thickness, thereby indicating a loss of homogeneity and uniformity in the coating layer.[11]

Water absorption

One-way analysis of variance (ANOVA) indicated a statistically significant difference in the water absorption values among the nine types of tested paper at a 5% probability level. The water absorption values across all treatments were categorized into nine distinct groups. As illustrated in Figure 4, the double-coated brown liner paper exhibited the lowest water absorption, while the highest values were observed in the control samples that were frozen for 4 months.

The water absorption of the double-coated brown cardboard decreased by 97.9%, compared to the control sample. One of the key properties of papers used in the packaging industry and other applications is their ability to act as a barrier against various liquids, particularly water. Water is a critical compound that can penetrate packaging materials and diminish food quality. Controlling water permeability is essential to prevent moisture transfer, which can negatively impact food quality during storage.

Table 4

Analysis of variance (F value and significance level) of the effect of variables on resistance

Properties	F
Thickness (µm)	11.472*
Water absorption (g/m^2)	44179032.8*
Surface smoothness (S)	9.143*
Contact angle (degree)	41354.970*
Burst index (kPam^2/g)	18.144*
Tear index MD (mNm^2/g)	2.127*
Tear index CD (mNm^2/g)	6.868*
Tensile index MD (mkN/m)	8.926*
Tensile index CD (mkN/m)	7.079*
RCT CM (KN/m)	4.464*
RCT CD (KN/m)	2.387*
Brightness (%)	954.123*

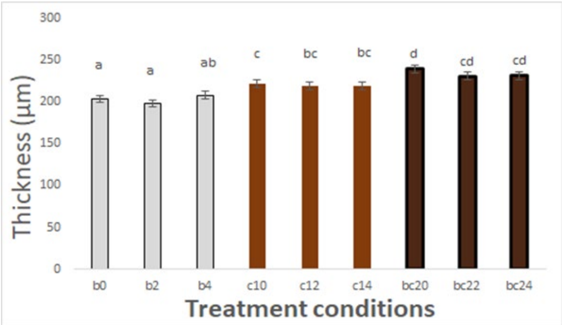


Figure 3: Mean thickness of differently treated paper samples

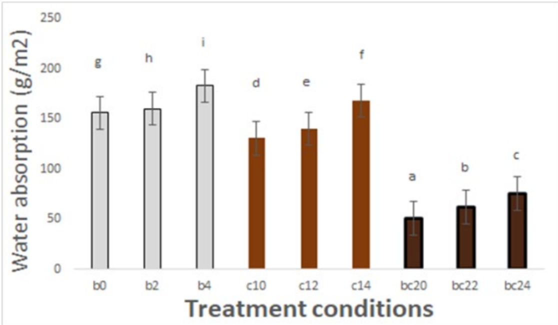


Figure 4: Mean water absorption of differently treated paper samples

The water absorption test, commonly known as the Cobb test, is one of the most important methods for assessing the resistance of materials used in food packaging and printing industries to water absorption.[12] Water permeability is a vital parameter in food packaging, and it can be improved through effective methods, such as coating the paper surface.[13] In general, the water absorption of paper depends on two main factors: the porous structure of the paper and the interaction between fibers and water. This test measures the amount of water absorbed when the paper is in direct contact with water. Increasing the coating on the surface of the paper reduces water absorption. Overall, the water absorption rate decreases with additional coatings, with a more significant reduction observed in the samples that were coated twice (b20, b22, b24). Among the twice-coated samples, sample b0, which was not subjected to freezing, exhibited the lowest water absorption rate.

Additionally, water absorption was reduced in both coated and frozen samples. In the paper network, water molecules are absorbed not only through the fibers, but also through the pores between the fibers and within the paper itself. Polymer coatings cover the paper's structure, filling the pores and forming a continuous layer. As a result, this layer or film significantly reduced water absorption.[14]

Furthermore, the hydrophobic properties of nano-polyurethane and nano-clay have significantly influenced the water absorption behavior of coated cardboards. Paper is a highly hygroscopic material; when humidity levels rise, it readily absorbs water, whereas it tends to release water when humidity decreases. Inside a refrigerator, as the temperature drops, humidity levels tend to increase. Consequently, papers respond to this rise in air humidity, leading to a further increase in water absorption rates over periods of 2 and 4 months.

The presence of coated biodegradable nano-substances has enhanced the adhesion of the surface of the paper. In addition to filling the thin areas and pores of the paper, these substances have formed a protective surface layer, resulting in a significant increase in water absorption resistance compared to the control sample. These findings are consistent with the results reported by de Lima Santos *et al*. and Tihminlioglu *et al*.[2,15]

Surface smoothness

One-way analysis of variance (ANOVA) revealed a statistically significant difference in the surface smoothness values among the nine types of tested paper at a 5% probability level. The surface smoothness values across all treatments were categorized into three distinct groups. As shown in Figure 5, the double-coated brown liner paper exhibited the highest surface smoothness, while the lowest values were observed in the single-coated liner paper samples.

The surface smoothness of the double-coated brown cardboard improved by 17.5% compared to the control sample. The smoothness of the paper surface is a key indicator of its printability. The use of double coating has contributed to an increased smoothness. It is important to note that this improvement is expected, and further enhancements can be achieved through additional treatments.[16]

Paper consists of a network of cellulose fibers that are bonded together. Despite undergoing various processes through rollers in the press and dryer of the paper machine, the paper retains many pores, resulting in surface unevenness or roughness. Filling these pores with coating materials effectively enhances the surface smoothness. The significant difference in smoothness observed is critical, as it directly affects the printability of the paper. The application of double coating has notably increased this smoothness, which is a normal outcome of the coating process.

Contact angle

One-way analysis of variance (ANOVA) revealed a statistically significant difference in the contact angle values among the nine types of tested paper at a 5% probability level. The contact angle values were categorized into nine distinct groups across all treatments. As illustrated in Figure 6, the highest contact angle was observed in the control sample and the samples frozen for 2 months, while the lowest values were found in the double-coated samples.

Tensile index

One-way analysis of variance (ANOVA) indicated a statistically significant difference in the tensile index values in the machine direction (MD) and in the cross-machine direction (CD) among the nine types of tested paper at a 5% probability level. The tensile index values for both MD and CD across all treatments were categorized into four distinct groups.

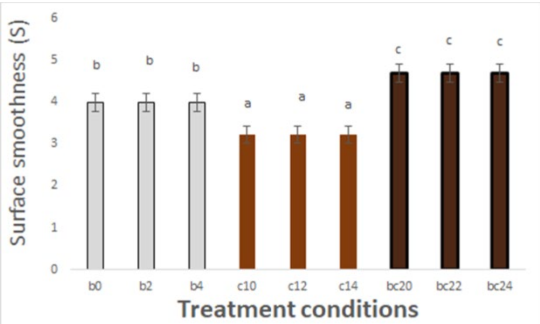


Figure 5: Mean surface smoothness of differently treated paper samples

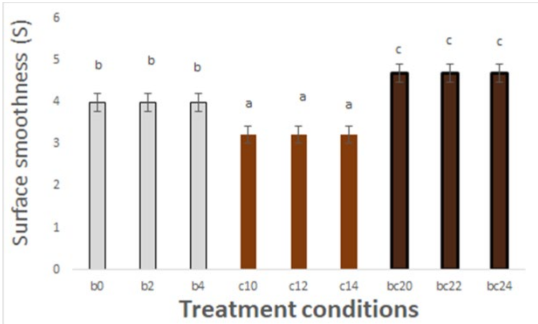


Figure 6: Mean contact angle of differently treated paper samples

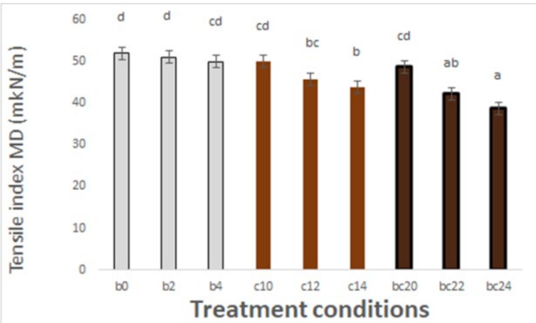


Figure 7: Mean tensile index in the MD of differently treated paper samples

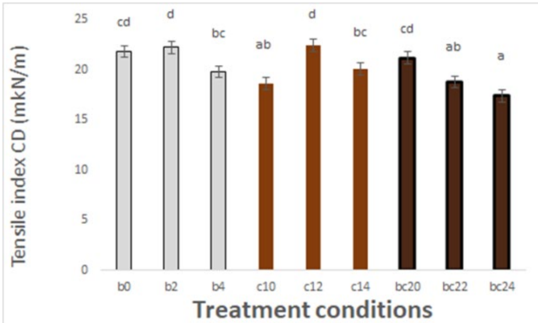


Figure 8: Mean tensile index in the CD of differently treated paper samples

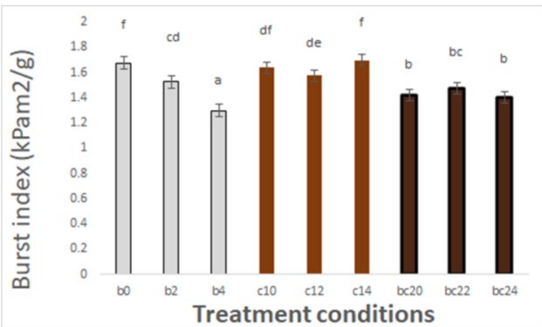


Figure 9: Mean burst index of differently treated paper samples

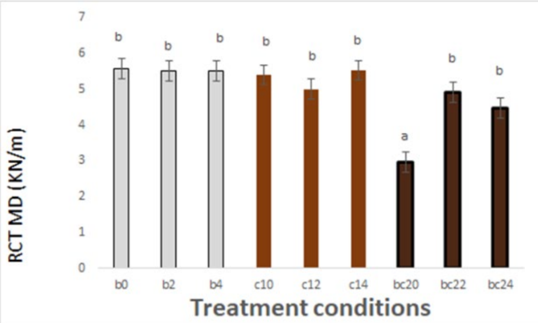


Figure 10: Values of mean ring crushing test in MD of differently treated paper samples

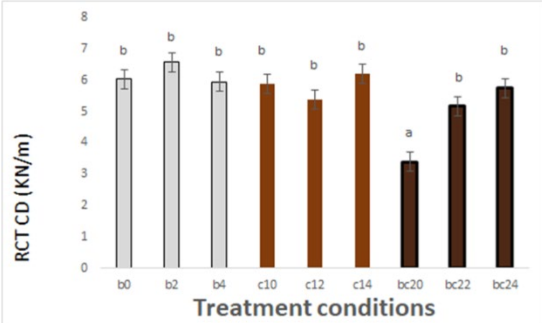


Figure 11: Values of mean ring crushing test in CD of differently treated paper samples

As shown in Figure 7, the highest tensile index in the machine direction was associated with the control sample, while the lowest values were found in the double-coated liner paper samples and those frozen for 4 months. Figure 8 illustrates that the highest tensile index in the CD was associated with the single-coated brown liner paper sample and the samples frozen for 2 months, while the lowest values were observed in the double-coated liner paper samples frozen for 4 months.

The tensile index in the MD of the double-coated brown cardboard decreased by 34.3% compared to the control sample. The tensile index in the CD for the double-coated brown cardboard decreased by 25.4% compared to the control sample. The tensile index is a more suitable measure of the overall bonding between fibers, representing a combination of various strength factors. The most significant influence on the paper's tensile index is the quantity and quality of fiber bonds. An increase in fiber bonding, achieved through enhanced refining or wet pressing, will lead to a higher tensile index for the paper. However, it is important to note that the tensile index of paper will always be lower than that of the individual fibers.

The reduction in the tensile index may be attributed to improper distribution of fibers within the coating matrix during preparation, as well as the inadequacy of the coating method, which can lead to stress concentration and a decrease in strength. Additionally, non-uniformity in the coating and the presence of uncoated areas may further contribute to reduced strength.

In the applied coating, the high percentage of nano-substances appears to shift the system's charge towards the negative side, causing the fibers to repel each other, consequently reducing the bonds between them.

Burst index and ring crushing index

One-way analysis of variance (ANOVA) indicated a statistically significant difference in the burst index values among the nine types of tested paper at a 5% probability level. The burst index values were categorized into six distinct groups across all treatments. As shown in Figure 9, the highest burst index was associated with the single-coated liner paper samples frozen for 4 months, while the lowest values were found in the control samples and those frozen for 4 months. The burst index of the double-coated brown cardboard decreased by 20% compared to the control sample.

One-way analysis of variance (ANOVA) revealed a statistically significant difference in the values of the ring crushing test both in the machine direction (MD) and in the cross-machine direction (CD) among the nine types of tested paper at a 5% probability level. The ring crushing index values in both directions were categorized into two distinct groups across all treatments. As illustrated in Figure 10, the highest ring crushing index in the machine direction was associated with the control sample, while the lowest values were observed in the double-coated liner paper samples. Figure 11 reveals that the highest ring crushing resistance in the CD was associated with the control sample frozen for 2 months, while the lowest values were found in the double-coated liner paper samples.

The burst index and the ring crushing index are crucial factors influenced by fiber length, fibers strength, and the bonding between fibers. While an increase in fiber length generally enhances these indices, the overall strength primarily depends on the quality of the bonds between the fibers. The incorporation nano-polyurethane and nano-clay has increased the coating thickness on the paper surface, which has reduced the bonding between the paper surfaces and the upper layers of the polymer, leading to a decrease in strength. Additionally, the introduction of nano-polyurethane and nano-clay results in a higher release of these substances during paper production, which diminishes bonding with the fibers and subsequently lowers the burst index and tear index of the paper. Another contributing factor to the reduced strength is the high concentration of positive charges created in the coating, which prevents the fibers from effectively approaching one another. This results in fewer bonding points between the fibers, reducing the paper's resistance to bursting and crushing. Moreover, the presence of nano-coating substances in the gaps between fibers leads to lower quality of inter-fiber bonds, resulting in a more significant reduction in strength, particularly in double-layer coatings. The ring crushing index of the paper in the MD of the double-coated brown cardboard decreased by 24.3% compared to the control sample. The ring crushing index of the paper CD for the double-coated brown cardboard decreased by 4.7% compared to the control sample.

Tear index in machine direction (MD)

One-way analysis of variance (ANOVA) revealed a statistically significant difference in the tear index values among the nine types of paper tested in both the machine direction (MD) and the cross-machine direction (CD) at a 5% probability level. For MD, the tear index values were categorized into three distinct groups across all treatments. As illustrated in Figure 12, the highest tear index in the machine direction was associated with the double-coated liner paper sample, while the lowest values were observed in the single-coated liner paper samples frozen for 2 months.

As regards the tear index values in the CD, these were categorized into four distinct groups across all treatments. As shown in Figure 13, the highest tear index in the CD was associated with the control sample frozen for 2 months, while the lowest values were found in the double-coated brown liner paper samples also frozen for 2 months.

The tear index in the MD of the double-coated brown cardboard reduced by 1.5% compared to the control sample. The tear index in the CD for the double-coated brown cardboard increased by 3.8% compared to the control sample. One of the key parameters affecting the tear index is fiber length. It also depends on the resistance between fibers and the strength of individual fibers. The presence of nanoparticles between the fibers typically weakens the bond between them, which reduces the resistance between fibers, while the resistance of individual fibers remains unchanged. Coatings have generally led to a reduction in the tear index, primarily due to the small dimensions of the nanoparticles. Their limited ability to create physical distance between the microfibrils of the fibers has had minimal impact on the overall bonding surface and, consequently, the resistance of the paper.

The tear index is crucial for assessing the strength of paper and cardboard, especially during the conversion stages when they are exposed to tearing stresses. Factors influencing paper tearing include fiber length, fiber diameter, and the number of fibers involved in the tearing process.[16]

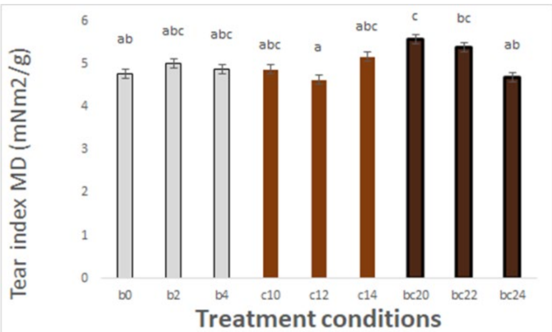


Figure 12: Mean tear index in MD of differently treated paper samples

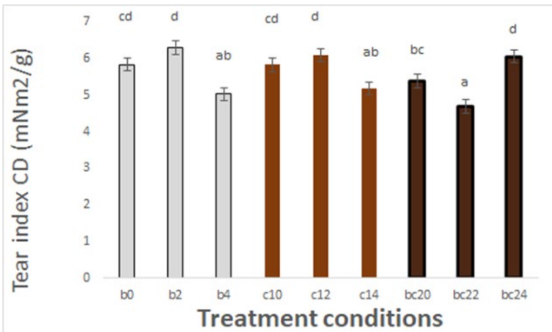


Figure 13: Mean tear index in CD of differently treated paper samples

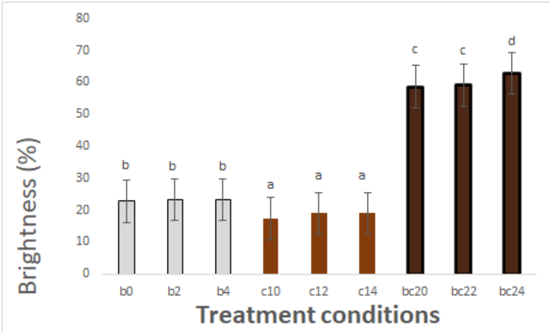


Figure 14: Mean brightness of differently treated paper samples

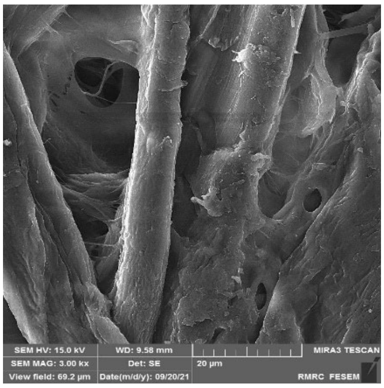
The bond between fibers is determined by the contact surface between them in the paper and the compounds in the pulp. In contrast, the intrinsic resistance of fibers increases with wall thickness and fiber diameter. With the use of coating substances, since these do not alter the morphological properties, such as fiber length and diameter, the tear index has not only remained stable, but also has increased, particularly in the machine direction.

Brightness

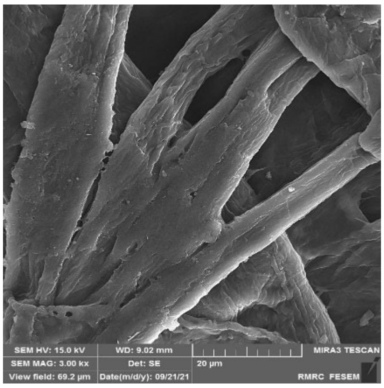
One-way analysis of variance (ANOVA) revealed a statistically significant difference in the brightness values among the nine types of tested paper at a 5% probability level. The brightness values were categorized into four distinct groups across all treatments. As illustrated in Figure 14, the highest brightness was associated with the double-coated liner sample frozen for 4 months, while the lowest values were found in the single-coated samples.

FE-SEM of paper structure

Figure 15 displays the FE-SEM images of the paper surfaces for the following samples: control Kraft liner brown paper (a), control Kraft liner brown paper frozen for 4 months (b), single-coated Kraft liner brown paper (c), single-coated Kraft liner brown paper frozen for 4 months (d), and double-coated Kraft liner brown paper (e).



(a)



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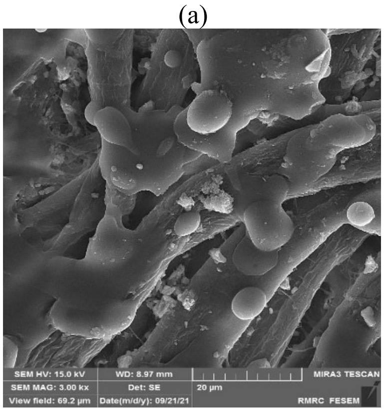

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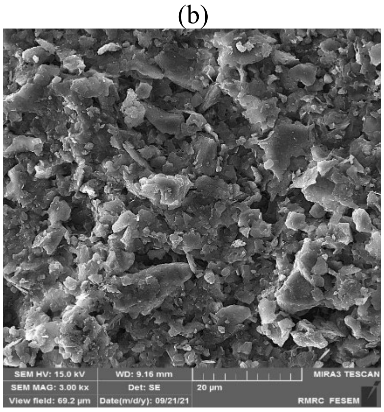

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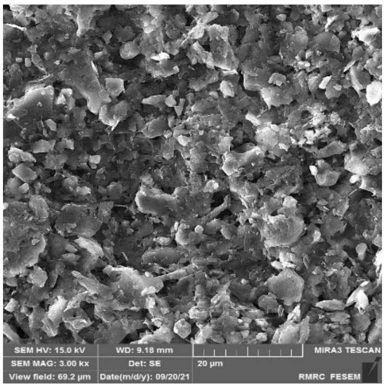

(e)

Figure 15: Paper surface FE-SEM images for (a) control Kraft liner brown paper, (b) control Kraft liner brown paper frozen for 4 months, (c) single-coated Kraft liner brown paper, (d) single-coated Kraft liner brown paper frozen for 4 months, and (e) double-coated Kraft liner brown paper

The untreated papers exhibited noticeable pores, while the coated samples showed very few pores.

The comparison of SEM images of the paper surface revealed that the coating solution, which contained nano-polyurethane and nano-clay, formed a polymer layer on the paper. This layer filled the pores and reduced the porosity of the paper. The observed non-uniformity in the coating was not due to inconsistencies in the coating itself, but rather resulted from the uneven porosity of the paper surface in different areas. This variation led to differing degrees of penetration of the coating substances into the pores.

In summary, the coating solution containing nano-polyurethane and nano-clay not only enhances the physical quality and reduces water absorption of the paper by limiting the surface pores, but also improves the surface characteristics and superficial properties of the paper. A significant advantage of using these coating substances is their high degradability, which is increasingly important in light of rising environmental concerns.[17] These substances are suitable substitutes for plastic materials and synthetic polymers because they can be easily recycled and decomposed in the environment.[18] Additionally, the use of these coatings extends the shelf life of food products and enhances their quality by providing a protective barrier layer. The double-coated samples exhibited very few pores.

CONCLUSION

The Kraft liner brown cardboard coated with nano-polyurethane and nano-clay has demonstrated significant improvements in various properties, notably water resistance and water absorption characteristics. The reduction in water absorption by 97.9% and the enhancement of surface smoothness by 17.5% indicate that this coating effectively fulfills the essential barrier requirements for packaging materials.

In our country, where plastic polymers are typically employed to enhance the barrier properties of packaging paper, these materials pose challenges for recycling. The biodegradable nature and recyclability of the nano-coated cardboard present a sustainable alternative to conventional plastic-based coatings, making it economically and environmentally advantageous for the packaging industry. Moreover, the reduced porosity and improved structural integrity suggest that this coating not only protects the contents from moisture, but also contributes to maintaining the quality of food products throughout their shelf life.

The findings highlight the potential of using nano-polyurethane and nano-clay coatings as viable substitutes for synthetic polymers in packaging applications, aligning with global efforts to reduce plastic waste and address environmental concerns. This innovative approach not only enhances the performance of packaging materials, but also promotes a more sustainable future in the packaging industry.

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