# Utilizing CMC/ZnO Blends Made at Various Mixing Ratios on Paper Surfaces and Their Impact on Paper Characteristics

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Surface treatments of paper and paper-based compounds are one of the most effective methods for improving different specifications of paper products, such as printability, paper strength values, absorption performance, and diminished surface roughness. In this study, cellulose derivatives of carboxymethyl cellulose (CMC) and zinc oxide (ZnO), an inorganic antibacterial material, were prepared in varying concentrations (1/1, 1/0.5, and 1/0.25) and applied to standard filter paper by the dip-coating method to examine the combined effects of these chemicals on paper properties and the performance of the investigation papers as a food packaging material. Density, dry-wet strength, Cobb values, oil-dye absorption, and air permeability of papers were investigated in addition to degradation tests, Fourier transform infrared spectroscopy, antibacterial activity, and scanning electron microscopy images. The density values of papers were determined between 0.421 and 0.468 g/cm$^3$. Although determined by different techniques, oil absorption and dye absorption performances showed similar patterns, and the addition of ZnO into the composition caused a decrease in the absorption performances. Dry strength and Cobb values increased with the ZnO addition, and strength values increased. Wet strength values. According to all of the findings, these papers would make excellent food packaging materials, particularly for dry, low-weight products.

*DOI: 10.15376/biores.20.3.7476-7494*

*Keywords: Surface treatment; Paper; Polymer; Paper coating*

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## INTRODUCTION

Over the years, paper and paper-based products have been used for different purposes, such as packaging material for different grades of materials, writing, and different specific purposes, such as microfluidics. During the papermaking process, different chemicals and specific agents are generally added to cellulosic suspensions to provide end products' specifications, from wet-strength polymers to fixatives. Due to the difficulty of retaining these chemicals in paper mats during the production process, surface treatments of paper are widely used for increasing paper specifications, especially in food packaging applications. Surface treatment of paper is one of the most effective specific applications to be given to the end paper product to obtain the desired properties, such as hydrophobic character, antibacterial performance, and fire retardancy. In general, the surface treatment process is followed by coating or surface sizing. In these, coating is a common method to improve paper's qualities. Especially it is possible to increase the hydrophobic specification of papers generally used in packaging applications, and the main aim in many cases is to increase the resistance of paper to air, water, or oil. The increasing hydophobic character of the paper being used in the packaging process clearly provides a way a way to keep packed food, vegetables, *etc.* stable for a long time. For this purpose, different chemicals were used, such as starch (Cao and Gao 2020; Xia *et al.* 2024), cellulose derivatives (Basta *et al.* 2015; Nizardo *et al.* 2024), nanocelluloses (Khan *et al.* 2013; Wang *et al.* 2024), chitosan (Khan *et al.* 2013; Tan *et al.* 2023), alginate (Priyadarshi *et al.* 2024), polylactic acid (PLA) (Abou-Zeid *et al.* 2018; Sundar *et al.* 2020), polyvinyl acetate (PVAc) (Choe *et al.* 2024), polycaprolactone (PCL) (Lo Faro *et al.* 2021), and polyhydroxyalkanoates (PHAs) (Tyagi *et al.* 2021). In addition to packaging, the coating process is also used to improve the surface printability and efficiency of the paper used in printing systems. Calcium carbonate, titanium dioxide, and kaolin are the most common inorganic fillers used for filling gaps between cellulosic fibers on the surface of paper, resulting in improved whiteness, opacity, and surface flatness. As a result, better printing performance is provided by coating.

Functional coating is a process that involves coating paper surfaces with specific materials instead of just inorganic fillers with adhesives. Through using specific coating materials, different features of papers can be improved, such as high antibacterial performance, catalytic activity, *etc.* (Li *et al.* 2020). Different coating techniques have been used, and all of them have both positive and negative effects. Extrusion coating (Basak *et al.* 2024), knife coating (Kunam *et al.* 2022), spray coating (Say *et al.* 2020), bar coating (Sundar *et al.* 2020; Park *et al.* 2024), and dip coating (Amorin-da-Silva *et al.* 2024) are the most common coating processes applied to paper surfaces.

A cellulosic ether produced by the reaction between cellulose and chloroacetic acid in an alkaline reaction medium, namely carboxymethyl cellulose (CMC), is being used in coating applications of paper due to its specific properties, such as its highly film-forming capacity with flexibility and transparency and its high level of barrier drainage against both oxygen and oil. Contrary to these positive specifications, due to the presence of high levels of OH and COOH groups in the CMC structure, CMC-based products' structure can be decomposed by the liquids. Nano-sized precursors are generally being used with CMC to improve the properties of CMC-based materials (Wang *et al.* 2019; Ramakrishnan *et al.* 2024).

As an inorganic nanomaterial, ZnO is one of the most common antimicrobial precursors, with a highly strict chemical structure and is used in many different industrial applications, ranging from photocatalysis to the paper industry. Specific characteristics of ZnO provide for using this material in different areas. Due to its high level of photochemical and optic specifications (3.37 eV energy band and 60 meV bond energy), ZnO has been used for producing electronic equipment. However, due to its chemical stability, low toxic properties, biocompatibility, and biodegradability, ZnO has also been used in producing textile and biomedical equipment (Martins *et al.* 2013; Kolodziejczak-Radzimska and Jesionowski 2014).

In this study, the cellulose ether (CMC) and one of the most common antibacterial agents (ZnO) were prepared by mixing different ratios of 1:1, 1:0.5, and 1:0.25 (weight ratio of CMC/ZnO), and this coating solution was applied to standard filter paper by dipping paper into the solution and bar pressing, respectively. The use of ZnO in CMC composition is justified by the study's primary goal of improving dry paper strength characteristics while maintaining antibacterial activity, and these mixing ratios were selected for investigating both CMC and ZnO's efficiency with high and low amounts in the composition. Densities of paper after absorption, dry and wet strength properties, COBB values, dye-oil absorption values, degradation rate of papers, antibacterial performances *versus E. coli* and *S. aureus*, FTIR spectra, and SEM images of papers were investigated.

## EXPERIMENTAL

### Materials

Standard filter paper for coating CMC/ZnO solutions was purchased from Schleicher & Schuell 5891 (grammage: 85 g/m², relative pore size: 12 to 25 µm). Coating components of CMC ($M_w$~250000 g/mole, DS: 0.7) and ZnO were purchased from Sigma Aldrich (Taufkirchen, Germany) and Kimyalab Chemical Company (İstanbul, Turkey). Model dyes of methylene blue (MB) and Congo red (CR) (1% stock solution) were purchased from TEKKİM Chemicals, Inc. (İstanbul, Turkey). Food grade sunflower oil was also purchased from the industrial market. Except for determining the wet strength of papers, deionized water was used during all experiments.

### Methods

*Preparing CMC/ZnO mixtures*

Stock solutions of CMC/ZnO were prepared by adding an appropriate amount of ZnO to the CMC solution to obtain a mixing ratio of 1/1, 1/0.5, and 1/0.25 (CMC/ZnO; w/w), and the mixtures were mixed by the high-shear mechanical stirrer for 24 h at 1200 rpm. Solution concentrations were adjusted to 1% for CMC (w/w), and pure CMC solution was also prepared at 1% concentration before preparing the mixing compositions.

### Coating CMC/ZnO Solutions onto Paper Surface

The surface coating process of papers was carried out using a simple method. Firstly, test papers were dipped into a CMC/ZnO solution, and a bar coated with silicone was applied to the paper surface to diffuse the solution homogenously. The excess part of the solution was removed from the top by a smooth, structured silicone slice. Finally, test papers were dried at 90 ºC for 30 min. All papers were balanced to calculate the absorption rate of the solution and stored until paper tests.

### Density Measurement

To determine the density values of papers, a fundamental method of converting volume fraction to weight fraction was applied. For this reason, coated papers were weighed, and the volume percentage was computed by using a micrometer.

### Absorption Amount of Coating Solution, Dyes, and Oil

The absorption amount (%) of the coating solution was performed to determine the weights of papers before and after coating performances. Oil absorption values were determined by placing 0.02 g of paper samples into oil (20 mL), and after 24 h at room temperature, the weight of the samples was determined using Eq. 1,

$$\text{Absorption (\%)} = \frac{[M_t - M_0]}{M_0} \times 100 \quad (1)$$

where $M_t$ is sample weight after coating or after 24 h (g) and $M_o$ *is* sample weight (g).

The paper's dye adsorption capacities were performed by a method belonging to Huang *et al.* (2019). After weighing the sample, it was placed in 10 mL of dye (0.5 g/L) and stirred at 200 rpm for 24 h. Dye concentrations were determined with a UV–vis spectrophotometer (Shimadzu UV Pharmaspec 1700) at different wavelengths, and adsorption capacities were calculated using Eq. 2,

$$\text{Dye adsorption capacity}\left(\frac{\text{mg}}{\text{g}}\right) = \frac{[C_0 - C_e]}{m} \times V \qquad (2)$$

where $C_e$ is the concentration of dye solution (mg/L), $C_0$ is the initial concentration of dye (mg/L), $m$ is the weight of the paper (g), and $V$ is the volume of the working solution (L).

## Degradation Extent

Extents of degradation of papers in water were measured. Briefly, 0.02 g of samples were put into 50 mL of water under room conditions. Samples were removed from the water at different times (1, 3, 6, 12, 24, 48, and 72 h) and fully dried. Dry weights were proportioned against the neat sample weight. Pure water is refreshed after each time zone, and decomposition rates were determined according to Eq. 3,

$$\text{Degradation extent (\%)} = \frac{[M_0 - M_t]}{M_0} \times 100 \qquad (3)$$

where $M_t$ is dry weight sample for each time zone (g) and $M_0$ is neat sample weight (g)

## FTIR Measurements

Chemical interactions between cellulose fibers and coating formulations were investigated by the FTIR diagrams, and experiments were performed in the 400 to 4000 $cm^{-1}$ spectrum with a resolution of 2 $cm^{-1}$ by the Bruker Alpha device.

## Dry-Wet Strength, COBB, and Air Permeability Performances

The tensile strength properties of papers were determined according to the TAPPI T404 om-87 (1987) standard with a Karl-Frank 800 Pendulum. Wet strength of papers was measured after immersion of paper strips into water for 0.5 min. The water absorption (COBB test) and air permeability (Gurley method-TAPPI T460 om-11) of papers were measured by using standards of TAPPI T441 om-98 (2013) and TAPPI T460 om-11 (2011), respectively.

## Antibacterial Activity Test

Bacterial resistance of papers was determined by using Gram-positive *Staphylococcus aureus* and Gram-negative *Escherichia coli* by means of the methodology of disk diffusion. Through using the switch technique, bacteria that had grown on enriched media for SS (*E. coli*) and BHA (*S. aureus*) were moved to agar media, where they proliferated across the agar medium. The inhibition zones of the medium associated with bacterial growth in the agar medium were assessed and averaged across many sites after a 24-h incubation period at 28 °C.

## SEM Images

The SEM images of papers were taken with an FEI Quanta FEG 450 (USA) at various magnifications to explore the micro-morphological structures. Cressington Spray

Coater (Ted Pella Inc., USA) was used to coat sample surfaces with gold-palladium (40 mA current, 50 mbar pressure) before imaging.

## RESULTS AND DISCUSSION

### Density and Absorption Values of Coating Solution

Figure 1 displays the density values for both the control and coated papers. The density value for pure paper was determined as 0.421 g/cm³; however, it is clearly apparent from the figure that the coating of paper resulted in an increase in density. The highest density increasing trend was observed for the composition of 1/0.5 as 0.4675 g/cm³. In general, density values were determined between 0.4210 and 0.4675 g/cm³. An increasing trend of density is an expected result for the raw material of filter paper. However, compared to the CMC/ZnO coating solution, a low level of ZnO (1/0.25) provided a lower level of density increment due to a low level of coating absorption, which can also be seen in Fig. 2. This situation can be explained by the filling of ZnO particles in gaps between cellulosic fibers on the surface of paper. In addition to density, grammage changes of the papers after the coating process can also be seen in Fig. 1. The graphic illustrates how changes in grammage exhibited the opposite pattern to changes in density. Different mixing compositions caused grammage to decrease compared to its density values. The control paper's grammage was determined to be 85 g/m², which was also given by the supplier, and the highest value belonged to the 1/1 composition at 85.45 g/m². This situation is an expected result and can be related to the coating absorption performances illustrated in Fig. 2. A modest decrease in grammage was induced by a higher ZnO content, and this circumstance also encourages a higher absorption efficiency of the 1/1 CMC/ZnO composition, which contains a higher amount of CMC.

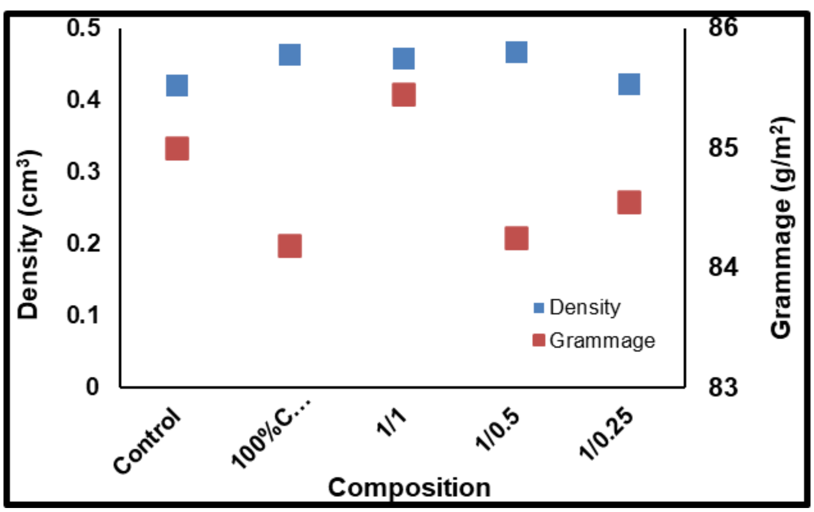


**Fig. 1.** Density and grammage values of neat paper and coated papers with different mixing ratios

Figure 2 shows the absorption amounts of coating solution on papers. The general trend shows that the addition of ZnO acts as a binding agent to the paper surface charged with negative –OH groups. Anionic groups presented on the surface of paper chemically interacted with cationic $Zn^{2+}$, and as a result, the coating solution easily coated the surface of the paper. Higher amounts of ZnO showed higher rates of absorption, which support this thesis. The highest absorption rate (3.76%) was observed by the composition of 1/1, which includes the highest amount of CMC and ZnO together. Increasing the absorption amount increased approximately twofold with the composition of 1/1 compared to pure CMC-coated paper. It is thought clearly that the binding effect of ZnO in the composition provided the diffusion of the CMC/ZnO solution onto paper easily.

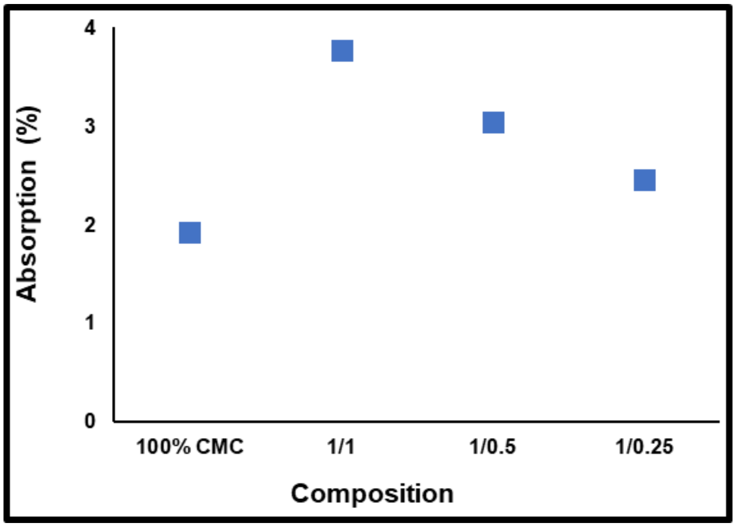


**Fig. 2.** Absorption amounts of coating mixtures by neat paper and coated papers with different mixing ratios

## Dye and Oil Absorption Performances of Papers

Figure 3 illustrates the dye and oil absorption performance of papers. When investigating MB dye, it is shown that all compositions prepared with CMC and ZnO had a lower degree of absorption compared to control paper (CP). However, coating with 100% CMC resulted in higher MB absorption, and the values were determined as 106.0%, 110.7%, and 89.0% for CP, 100% CMC, and 1/0.25 compositions, respectively. Due to the highly hydrophilic nature of CMC, higher absorption with compositions that have a higher percent of CMC are expected results. The hydrophilic nature of both cellulosic fiber presented in paper and CMC provided higher absorption of dye. Similar performances were also determined for CR absorption. The CP's absorption was determined as 87%, and the highest performance was provided by the 100% CMC as 100.9%, similar to MB results. The minimum CR absorption of all compositions belonged to 1/0.25 CMC/ZnO. As a result of dye absorption performances, it can be clearly stated that increasing amounts of ZnO in composition affect the dye absorption negatively. This situation can be explained as a hydrophobic nature of ZnO by Roy and Rhim's (2020) study in which CMC/curcumin/ZnO mixtures of the films were produced. According to results, the addition of ZnO into the CMC composition increased the water contact angle from 43.2 to 54.7 for the pure CMC film and CMC/ZnO composition, respectively. Jabin *et al.* (2019) produced CMC/ZnO-based composites with different compositions to investigate the dye-removing efficiency. Different parameters were investigated for the removal of cationic sapphire blue dye, such as dye concentration, dosage amount, time, and pH, and obtained results showed that all CMC/ZnO compositions with different proportions had higher dye-removing efficiency compared to neat CMC composites. When comparing the two dyes, higher MB absorption was determined by 100% CMC, and this situation is probably caused by the highly chemical interaction between the carboxyl groups of CMC and the cationic sites of MB. The decreasing trend with increasing amounts of ZnO that is the cationic character of the chemical also supports this suggestion. Higher amounts of CMC in a 1/0.25 composition showed a higher degree of MB absorption, parallel to this issue. However, anionic CR behaved opposite to MB due to anionic sites' interaction with $Zn^{2+}$, which increased the amount of ZnO in the composition and had a higher level of absorption. One of the other supportive studies about this situation was performed by Priyadarshi *et al.* (2020), in which CMC/ZnO hydrogels were produced, and according to the study, water absorption performances were affected negatively by increasing ZnO in the CMC composition. This phenomenon was explained by the crosslinking of $Zn^{2+}$ and CMC' -OH groups that resulted in a compact structure, and water could not penetrate into the inner part of the composition.

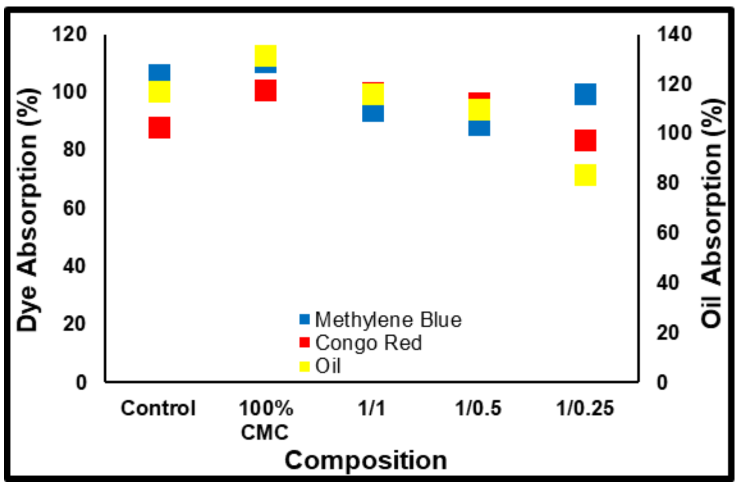


**Fig. 3.** Dyes (MB and CR) and oil absorption performances of neat paper and coated papers with different mixing ratios

Oil absorption performances of coated papers are also shown in Fig. 3. The highest oil absorption value was provided by the 100% CMC-coated paper, which has a highly hydrophilic surface to liquids. It is also apparent that a higher amount of ZnO in the composition caused a decrease in oil absorption. Sunflower seed oil comprises different chemical groups, but the main chemical structure is introduced by acidic precursors such as oleic acid, linoleic acid (saturated), palmitic acid, and stearic acid (unsaturated) (Simpson *et al.* 1989; Hu *et al.* 2001; Jalilian *et al.* 2012). All these acids have anionic carboxyl fractions, and probably cationic $Zn^{2+}$ ions react with anionic sites of these fatty acids, resulting in high oil absorption seen for 1/1 and 1/0.5 compositions. However, 1/0.25 composition coated paper, which have a higher amount of CMC, provided a lower degree of oil absorption. This situation can be explained by the Gurley Method (TAPPI T460 om-11) results of paper, which gives information about air permeability of paper (Fig. 8). The addition of ZnO into CMC hydrophilic CMC caused a decrease in the hydrophilicity of the composition and also filled the gaps between cellulosic fibers. As a result, diffusion of oil into the Z axis of the paper diminished.

## Degradation Ratio of Papers

Degradation ratio values of papers were determined for 72 h to investigate the efficiency of CMC/ZnO coating to prevent the decomposition of paper. In Figure 4, degradation values are shown as % depending on time. It is apparent from the Fig. 4 that decomposition was stabilized at 48 h, and all papers' degradation remained stable and did not show any change after this point. The maximum degradation of paper was for the 1/1 CMC/ZnO composition, and the lowest degree of degradation was provided by the control paper. This case clearly demonstrates how, depending on the duration, the CMC/ZnO coating process can be eliminated from the surface even at low levels. However, it should be stated that the maximum degradation rates of all samples were seen at almost low levels. The maximum degradation of the 1/1 composition was determined to be only 2.99% for 6 h. The degradation of all papers except the control paper, most probably were caused by the ZnO due to a low level of hydrophilic nature. While CMC binds on the paper surface easily by H bonding with cellulose fibers, ZnO is removed from the structure, which contributes to the observed degradation. This situation was also observed in a study by Rakhshaei and Namazi (2017). In this study, CMC-based hydrogels were prepared for wound dressing material using TC (tetracycline)-loaded ZnO-MCM-41. According to erosion studies, the addition of ZnO into CMC caused a high degree of erosion, approximately 100%, and this situation was explained as the reaction of ZnO with protons under an acid medium, resulting in the dissolution of ZnO.

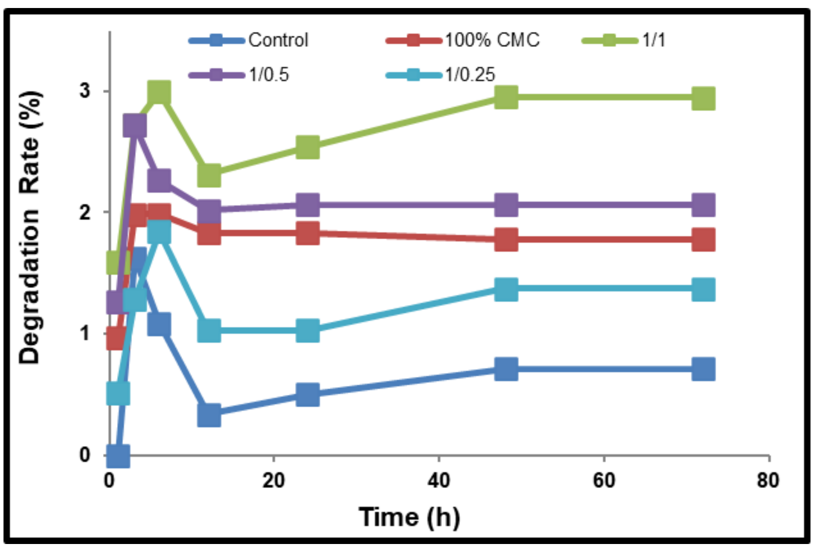


**Fig. 4.** Degradation ratios of neat paper and coated papers with different mixing ratios in water

## FTIR Diagrams

Figure 5 displays the FTIR patterns for both surface-treated and control papers. When investigating the control paper's FTIR spectra, some characteristic peaks can be seen clearly. In general, peaks between 3000 and 3750 $cm^{-1}$ can be generally attributed to O-H groups' stretching vibrations, and peak 3340, which is seen as 3333 $cm^{-1}$ in this study, is associated with OH' groups chemical activities, as reported in a study by Sathishkumar *et al.* (2013). The peaks of 2899 $cm^{-1}$ seen as 2895 and the peaks of 2129 $cm^{-1}$ and 2019 $cm^{-1}$, which are seen at 2122 $cm^{-1}$ and 2011 in this study, are related to C=C stretching vibrations. However, the peak at 1635 $cm^{-1}$, which moved to 1645 $cm^{-1}$ in this study, is related to the adsorbed water, as explained in a study by Liu *et al.* (2011)

and Md Salim *et al.* (2021). The peaks present at 1427, 1364, 1334, 1159, and 894 cm$^{-1}$ in this study are assigned as stretching and bending vibrations of $CH_2$ and -CH, -OH, and C-O bonds in cellulose, respectively (Xu *et al.* 2013; Hospodarova *et al.* 2018). The same image displays clean FTIR patterns for CMC. The biopolymer's distinctive peaks are located at 1586 cm$^{-1}$, 1412 cm$^{-1}$, and 1019 cm$^{-1}$. Asymmetric stretching vibrations of the carboxyl groups, symmetrical carboxyl moieties, and C-C stretching of CMC are linked to these, respectively (El Miri *et al.* 2015; Roy and Rhim 2020). Although there were minor variations in the peaks with decreasing intensities, the FTIR spectra of surface-coated and control papers behaved nearly identically. It can be seen from the 100% CMC-coated paper that neat CMC's characteristic peak of 1586 cm$^{-1}$ disappeared. This situation may be due to a high ratio of chemical interaction between carboxyl groups and water, which was used for the preparation of the CMC stock solution. The anionic carboxyl fraction probably reacted with $H^+$ ions of water molecules, and as a result of this phenomenon, the main characteristic peak of CMC was removed. Additionally, carboxyl fractions of CMC can easily be seen for both CMC/ZnO compositions. One of the most obvious changes seen from the FTIR spectra is that the addition of ZnO into CMC composition with different dosages caused a decrease in carboxyl efficiency, and CMC's characteristic peak of 1586 cm$^{-1}$ moved to 1549 to 1550 cm$^{-1}$ for 1/1 and 1/0.25 mixing rated compositions with small intensity. One of the other important changes that can be seen from the CMC/ZnO compositions FTIR patterns is the changing band from wide to narrow with the addition of ZnO into CMC. A small and strong structure replaced the broad band observed for pure CMC between peaks 2878 and 3557 cm$^{-1}$. Liu *et al.* (2016) clarified this problem by stating that coordination bonds were developing between ZnO and CMC functional groups. Additionally, it is seen clearly from the Fig. 5 that different mixing ratios of CMC/ZnO compositions did not cause any chemical adverse effect on the paper surface.

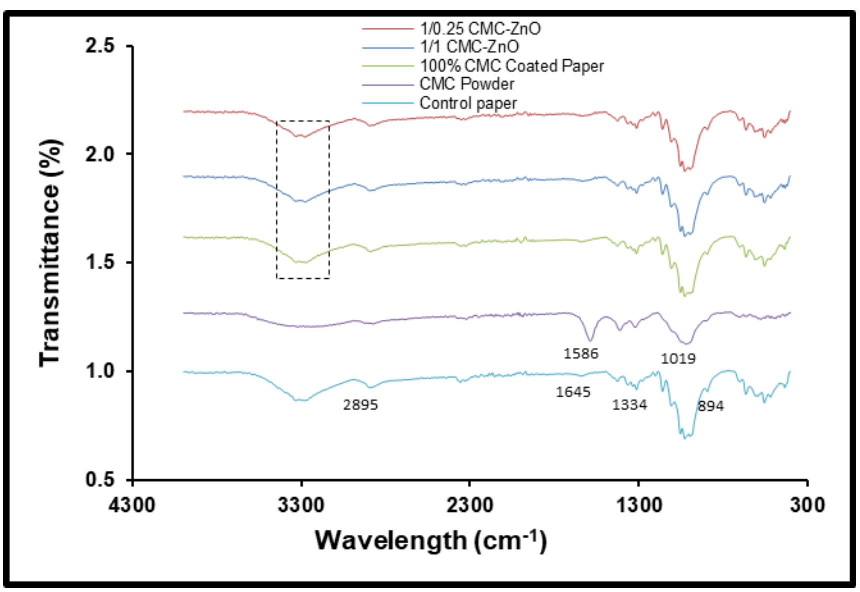


**Fig. 5.** FTIR spectra of control paper, CMC powder, 100% CMC, 1/1 CMC/ZnO and 1/0.25 CMC/ZnO coated papers

## Dry-Wet Strength, COBB Values, and Air Permeability Performance of Papers

The efficiency of CMC/ZnO compositions and pure CMC on paper strength values was investigated by determining the paper's dry and wet strength values. Dry strength performances of CMC/ZnO-coated papers showed higher strength values compared to CP. The maximum strength value belonged to 100% CMC-coated paper, determined as 365 Nm/g, which shows an increase in paper strength as 71%. The second highest value was observed by the composition of 1/0.25, similar to 100% CMC-coated paper as 364 Nm/g, which had a higher level of CMC. It is clearly apparent from the Fig. 6 that CMC promoted increased dry strength values, but the wet strength values were diminished when ZnO was added. CMC is one of the most commonly used cellulosic additives in the paper industry, as a strength agent, color stabilizer, fire retardant, and also a surface smooth enhancer (Rahman *et al.* 2021).

Increasing paper strength by adding CMC has been studied by different researchers. Fatehi *et al.* (2010) proposed a study in which modified chitosan and CMC with different molecular weights were investigated for increasing paper properties. A 1:1 charge ratio-based modified chitosan with high molecular weight CMC showed the highest values of tensile, burst, and tear indices. A parallel result with this study was presented by Basta and El-Saied (2008), in which CMC-Cu(II) complexes using different Cu(II) precursors with CMC were used as paper additives. According to results, CMC/$CuSO_4$ complexes showed higher performance from pH 3.5 to 5 for breaking, tearing, and bursting properties. Additionally, the increased strength properties of paper with the addition of CMC was presented by different researchers (Strand *et al.* 2017; Basu *et al.* 2021). It is evident that the dry strength decreased when ZnO was added to the CMC composition. Conversely, a lower concentration of ZnO (1/0.25) in the CMC composition produced a higher degree of dry strength value, demonstrating the high efficiency of CMC in dry strength attributes. The dry strength of paper with a higher concentration of ZnO in the coating solution is believed to be reduced *via* a variety of mechanisms. One of these is that the presence of ZnO nanoparticles on paper surfaces probably forms weak points that cannot show resistance to breaking force during the tensile strength test. Another issue is thought to be that chemical interactions between ZnO's cationic $Zn^{2+}$ ions with anionic carboxyl fractions of CMC diminished its efficiency on paper. It can be seen from Fig. 6 that 100% CMC resulted in the highest dry strength performance. When ZnO was added to CMC stock, the coating solution's absorption amount on the paper decreased, and as a result of the lower retention, a lower dry strength value was observed.

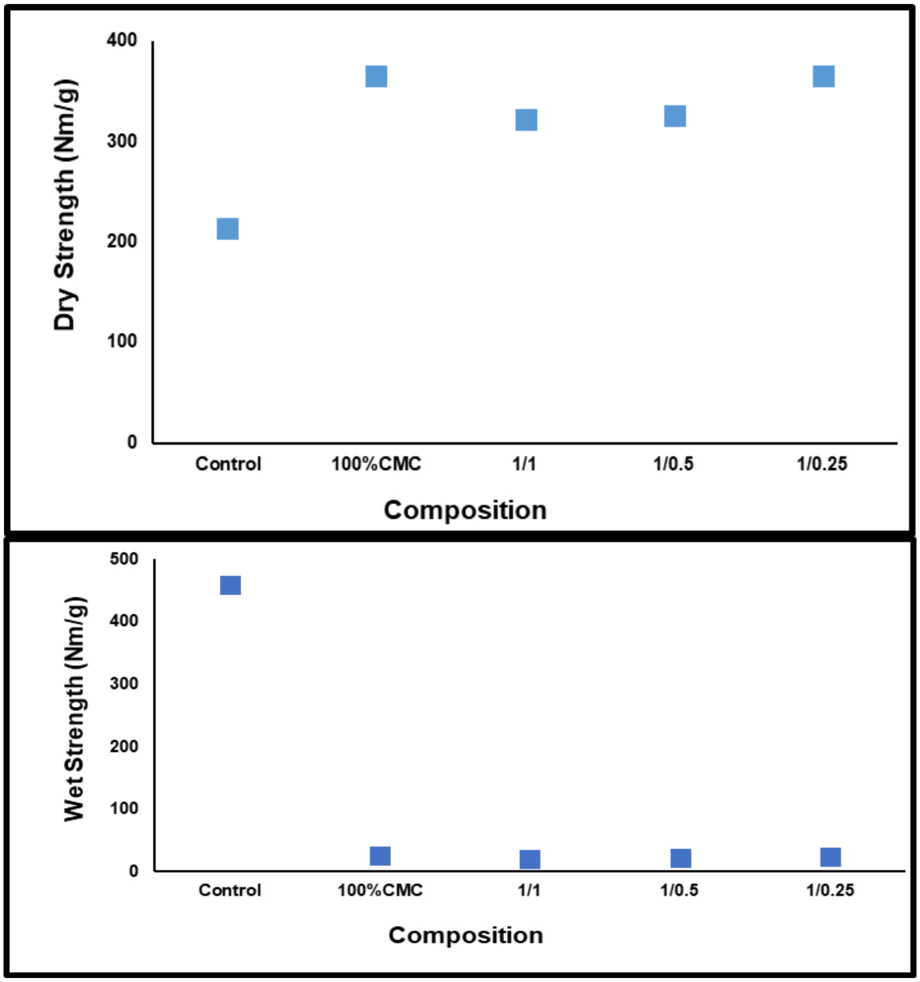


**Fig. 6.** Dry and wet strength values of neat paper and coated papers with different mixing ratios

Figure 6 displays the wet strength values of both neat paper and surface-coated papers. Coating the paper surface with CMC and CMC/ZnO compositions caused a negative effect on the wet strength of the papers. Wet strength values decreased from 459 Nm/g to the lowest degree of 20 Nm/g with the composition of 1/1 CMC/ZnO. The 100% CMC demonstrated a slightly better degree of wet strength than ZnO-added composites, despite the fact that the values were almost identical. Although the addition of CMC provided an increase in dry strength properties, wet strength values were negatively affected. This situation is expected, and the highly hydrophilic groups' presence, such as OH and COOH in CMC, is responsible for this issue with high probability. Weak spots and broken H bonds following water molecule penetration into the fiber mat led to a low resistance compared to liquid. However, one of the other issues about low performance for wet strength is that non-dissolved ZnO particles in the composition caused weak points on the fibers, and after treating with water, non-bonded ZnO particles probably separated through the paper surface, resulting in a high number of weak points.

Results of papers' Cobb test performances, which give information about the water uptake of the paper, are illustrated in Fig. 7. According to Fig. 7, surface coating of paper with only CMC and CMC/ZnO compositions clearly increased the water uptake performance of papers. The highest performance was provided by the 100% CMC-coated paper as 136.5 g/m², which shows increasing water uptake of 71.3% compared to neat paper. However, the addition of ZnO caused a decrease in water uptake, but it should be pointed out that all compositions applied onto the paper surface provided higher performance compared to the neat paper. The very hydrophilic property of the CMC polymer, which reacts with water molecules by enhancing H bonding, is assumed to be the cause of the increased Cobb values, particularly those brought about by CMC addition. Additionally, increasing specific surface area with cellulose fibers and CMC also provided higher water uptake performances. The CMC's water uptake performances were studied and cited by different researchers. Laine *et al.* (2002) investigated the strength properties of papers prepared by bleached kraft softwood pulp modified with CMC by surface application, and obtained results showed that surface modification of fibers by CMC increased the water retention value, and a similar result with this study was presented by Liimatainen *et al.* (2009).

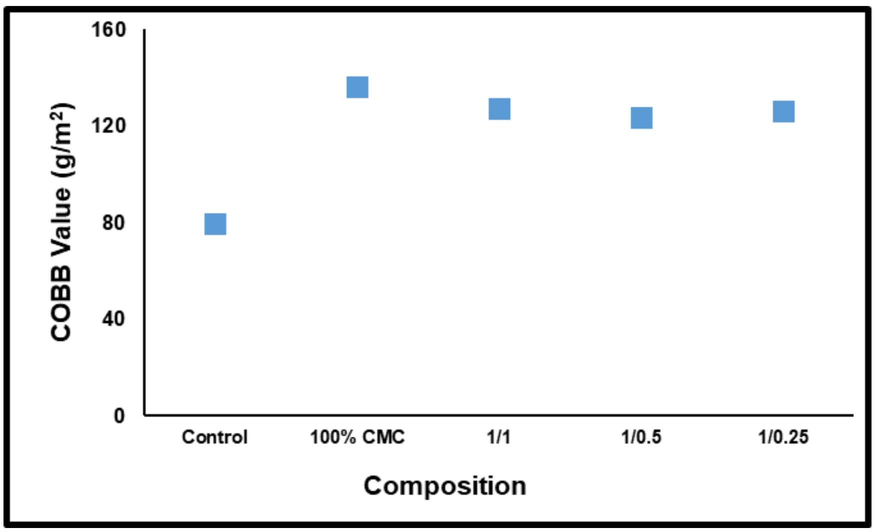


**Fig. 7.** Cobb values of papers neat paper and coated papers with different mixing ratios

Figure 8 displays the air permeability results of the papers. Similar to Cobb values, coating papers with only CMC and CMC/ZnO compositions provided higher air permeability results compared to CP. The maximum air permeability was obtained by the paper that was coated with 100% CMC, and the determined value was 0.85 s/100 mL. However, the addition of ZnO into the CMC matrix with increasing amounts caused a decrease in the permeability. Different researchers have examined with the effects of CMC on the performance of the coated paper. Mortazavi *et al.* (2021) investigated the CMC-nanomineral coating solutions' effects on the paper properties. According to results, air permeability properties were negatively affected by increased CMC compared to neat paper. Wang *et al.* (2022) reported that carboxymethyl chitosan/sodium alginate/ carboxymethylcellulose-based coating formulations were prepared, and air permeability and oil resistance of the papers were investigated. Results showed that air permeability and oil resistance performances were affected directly by the coating weights. Different coating weights were measured, and after 4.08 g/m², air permeability remained stable. In the present study, air permeability performances were directly correlated with the absorption of the coating solution, as can be seen in Fig. 2. The similar patterns of absorption with air permeability results can be seen clearly, and it is obvious that the addition of ZnO into the CMC composition caused the lower absorption rate and also lower air permeability results. This situation can be thought of as a result of the chemical interaction between the carboxyl fraction of CMC and $Zn^{2+}$ ions present in ZnO, and this interaction probably caused polymer blocks to come closer. As a result of this, many more microgaps were present on the paper, and air could move easily throughout the paper.

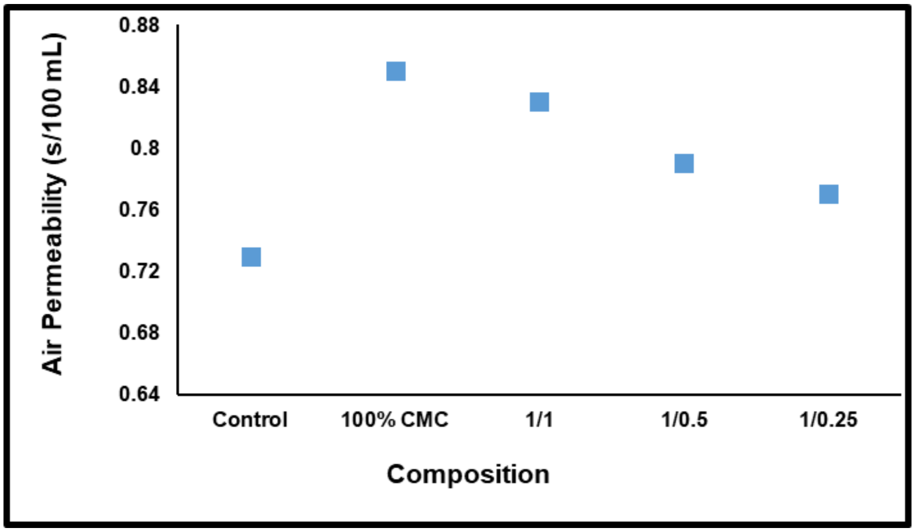


**Fig. 8.** Air permeability values of neat paper and coated papers with different mixing ratios

## Antibacterial Activity of Papers

Bacterial resistance of papers with neat and CMC/ZnO-coated samples was investigated by the simple disc diffusion method, and inhibition zone sizes are seen in Table 1. It can be clearly seen that antibacterial performances were determined for *S. aureus* and *E. coli* with CMC/ZnO compositions. However, CP and 100% CMC surface-treated paper showed no inhibition zone. The increasing amount of ZnO in the CMC/ZnO composition increased the antibacterial efficiency of the paper *versus* both *S. aureus* and *E. coli*. In general, pure CMC has no antibacterial performance against *S. aureus* and *E. coli* bacteria (Han and Wang 2017). However, CMC-based products having antibacterial performance are generally prepared using different antibacterial agents, such as different metallic compounds (ZnO, CuO, $TiO_2$, *etc.*), or biopolymers, such as chitosan (Yadollahi *et al.* 2015; Hu *et al.* 2016; Roy and Rhim 2020). In a study prepared by Roy and Rhim (2020), CMC/ZnO-based films were produced using curcumin and ZnO as antioxidant and antibacterial agents, respectively. A bacterial resistance test was performed using *E. coli* and *L. monocytogenes* bacteria. According to results, curcumin and ZnO-added films showed antibacterial performances for both bacteria; however, pure CMC film showed no bacterial resistance. Furthermore, the addition of ZnO to CMC polymer demonstrated greater efficiency for *E. coli* bacteria compared to *L. monocytogenes*. CMC/ZnO-based nanocomposite hydrogels were produced by Yadollahi *et al.* (2015). According to antibacterial performance tests, increasing CMC/ZnO concentration provided a wider inhibition zone for both bacteria of *S. aureus* and *E. coli*. Hydrogel having the highest amount of ZnO coded as Z4 showed the highest antibacterial performance. Parallel to these studies, antibacterial performances of papers coated with CMC/ZnO mixtures are expected. The main mechanism of the antibacterial activity of ZnO is not clear, but it is stated that free Zn ions present around the bacteria cause structural deformation of the cell membrane of the bacteria, resulting in increased permeability. As a result of the permeability change, Zn ions can reach and diffuse into the cell wall easily and disrupt the cell wall, resulting in the death of the cell (Xie *et al.* 2011; Roy and Rhim 2020).

**Table 1.** Inhibiton Zones of Papers After Antibacterial Test

| Sample | Inhibition Zone (mm) *S. aureus* | Inhibition Zone (mm) *E. coli* |
|---|---|---|
| Control | - | - |
| 100% CMC | - | - |
| 1/1 | 25.3 | 21.1 |
| 1/0.5 | 20.08 | 18.6 |
| 1/0.25 | 16.24 | 11 |

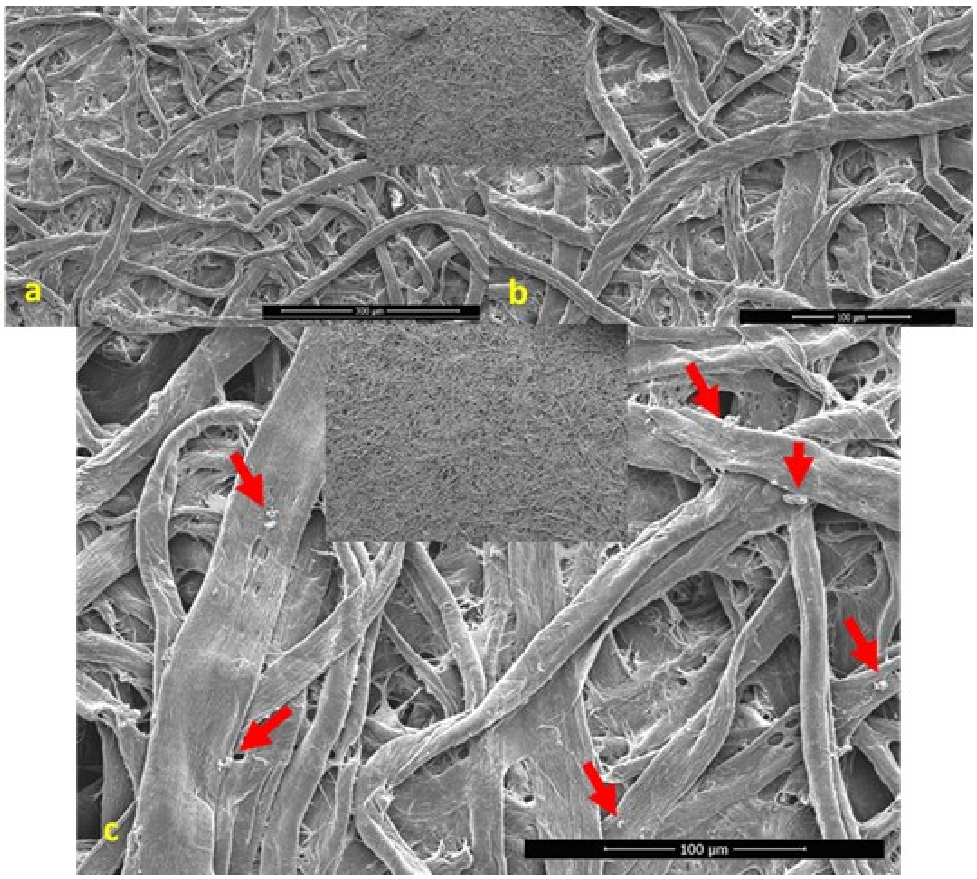


**Fig. 9.** SEM images of control paper (a, b) and CMC/ZnO (1/0.25) (c) papers

## SEM Images of Papers

The SEM images of neat paper and 1/0.25 composition coated paper with magnifications of 500X and 1000X are shown in Fig. 9 (a through c). Figures 9 a through 9b shows the control papers' surface microimages, and seen as interconnected fibers, they presented the paper mat. A small view of the microstructure of paper shown in the top of the Fig. 9a-b shows a bit of coarse structure with different-sized gaps. However, the highest dry strength values of all CMC/ZnO compositions were obtained by the 1/0.25 CMC/ZnO composition, and SEM images of this paper can be seen in Fig. 9c. Low levels of ZnO nanoparticles are shown through paper mats labeled with red arrows. In association with SEM images, presenting a low amount of ZnO nanoparticles provided a low level of antibacterial effect *versus S. aureus* and *E. coli* bacteria. When compared to the micromorphological surfaces of control and 1/0.25 CMC/ZnO coated paper, the coating efficiency of CMC is clear. From the perspective of the Fig. 9c, it is evident that CMC functions as a filler between fiber blocks for 1/0.25 CMC/ZnO-coated paper. As a result of CMC's blockage effect, disorganized gaps present between fibers were blocked, and a smooth, soft paper surface was obtained. During the study, CMC and CMC/ZnO-based mixtures were easily applied to paper surfaces without any curling/wrinkling after the whole process, and this situation is explained in a study prepared by Shankar and Rhim (2018) as a high compatibility between paper and biopolymer's high hydrophilic nature.

## CONCLUSIONS

Carboxymethylcelullose (CMC)/ZnO coating formulations were applied to standard paper surfaces to investigate different properties of paper. Density, dry-wet strength, water uptake, air permeability, and degradation performances were investigated with Fourier transform infrared (FTIR) experiments and bacterial resistance performances.

1. The obtained results showed that coating of paper increased the density values, and a higher amount of ZnO in the CMC matrix caused a lower degree of absorption of coating solutions, resulting in a lower increase of grammage. The findings of dye absorption performance showed that the addition of ZnO to the CMC composition decreased the absorption amount, with the exception of MB (1/0.25) performance; nevertheless, the lowest degree of ZnO (1/0.25) boosted MB absorption.
2. Dry strength and Cobb values were increased by all CMC/ZnO compositions compared to the control paper (CP), and the highest performances were obtained by the 100% CMC coated papers. Contrastingly, wet strength performances of the paper were affected negatively by all compositions and also with 100% CMC.
3. The CMC/ZnO compositions with different proportions showed antibacterial performance *versus* both Gram-positive and Gram-negative bacteria, and no inhibition zones were obtained by the control paper and 100% CMC. All of the findings indicated that these papers could be a viable option for food packing, particularly for dry, low-weight items.

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Article submitted: March 19, 2025; Peer review completed: April 19, 2025; Revised version received: April 28, 2025; Accepted: July 3, 2025; Published: July 22, 2025.
DOI: 10.15376/biores.20.3.7476-7494