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INVESTIGATION OF BIO-BASED PAPER COATINGS CONTAINING NATURAL PIGMENT AND THEIR HEAT RESISTANCE PROPERTIES

Arif Ozcan [1], Dogan Tutak [1], Lutfi Ozdemir [2], Omer Bunyamin Zelzele [2]
[1] Marmara University, Faculty of Applied Sciences, Printing Technologies, Istanbul, Turkey
[2] Marmara University, Vocational School of Technical Sciences, Printing and Publishing Technologies, Istanbul, Turkey

Abstract: *Cellulosic paper which is created from cellulose-rich wood fibers has a highly flammable property and can easily catch fire. Some studies are being conducted to improve and increase the flame-retardant properties of paper, but there are currently very few studies that apply flame retardant coatings on sheet paper. By coating the paper surface using some bio-based coating materials containing natural pigments, oxygen can be prevented from reaching cellulose and the rapid ignition of the paper can be delayed. In this study, paper coating materials were prepared using three different types of pigments (PCC, zeolite and bentonite) and coated on 80 g/m^2 base paper. Thermogravimetric analysis (TGA) measurements were performed on the coatings. Prints were made on the coatings with an Epson Stylus Photo R3000 digital printing machine, and printing color measurements were made using an X-Rite eXact spectrophotometer. The surface morphology of the coatings was examined, and the limited oxygen index (LOI) was determined. As a result of the investigations, it has been determined that the coatings created increase their flame retardancy and have a good hydrophilia. At the same time, when the printing parameters were examined, it was determined that the measured values were in accordance with ISO 12647-2 standards.*

Key words: paper coating, inkjet printing, printability, heat resistance

1. INTRODUCTION

Paper is widely used for magazines, journals, valuable documents, and packaging due to its biodegradability, easy recyclability, high sustainability, and low cost (Zhang et al., 2021; Zeng, Gu & Cao, 2020). Despite these positive features, papers have poor barrier properties against water, oil, and oxygen (Nechita & Roman, 2020). In order to improve the weak barrier properties, attempts are made to improve this weak barrier property by using polymers, aluminum foils and different waxes. However, these coatings used consist of fossil-based synthetic materials that are difficult to biodegrade, especially in recycling (Kabir et al., 2020).

Sustainable biopolymers have become a good alternative to petroleum-based plastics in recent years. Among the biopolymers, proteins, polysaccharides, lipids, and polyesters can also be used to produce films and coatings (Lisitsyn et al., 2021).

It is common to treat the paper surface to obtain better substrate performance. Surface treatments improve optical and mechanical properties. With these treatments, certain pigments and binders are applied to the surface according to specific formulations. Starch derivatives and polyvinyl alcohol are common binders, and calcium carbonate and clay are also the most used pigments (Gadhave, Gadhave & Dhawale, 2022).

However, paper, which consists of cellulose fibers, is an extremely flammable and combustible material. For this reason, many valuable works have been destroyed in fires. These losses have caused the loss of important information for human history. For example, the great fire in the Notre Dame Cathedral in Paris in 2019 caused many works to be destroyed by fire. Applications that are applied to papers during or after production and that give them fireproof properties have become quite widespread. These applications increase the resistance of papers to burning and improve their flame retardancy. This is extremely important for the safe and long-term preservation of valuable works of art (Fang et al., 2022).

In the printing industry, many products with high heat resistance are required to be produced (Ozcan et al., 2020). In recent years, many methods have been used to impart fire retardant properties to papers. These can be listed as dipping, coating, spraying and layer-by-layer (LBL) bonding. The most effective method is the coating method. However, halogen and phosphorus-containing components frequently used here are extremely dangerous for the environment (Fang et al., 2022). Halogen-based flame retardants have been reported to cause cancers and poor neurocognitive functioning in children (Chen et al., 2020).

For this reason, the use of natural, environmentally compatible, and environmentally friendly pigments as fire retardants is increasing. There are many pigments used as natural coatings. Zeolite is a natural pigment with unique crystal structures and is also used in the paper industry. Thermal stability and catalytic activity are just two of its distinctive features. It is mostly synthesized from sodium, aluminum and silicon sources and made ready for use (Soares et al., 2023). Easy availability, low cost, penetrability, low refractive index, and compatibility with other materials have made calcium carbonate ($CaCO_3$) one of the most widely used pigments in the paper industry (Ma et al., 2022). Bentonite clay is one of the materials that human beings have used in their daily lives for centuries (Borah, Nath, & Saikia, 2022). This natural pigment has many properties and is widely used in agriculture and paper industry (Wang et al., 2023). It is also used as a filler in paper production or later as a coating material (Umar et al., 2022).

The aim of the research is to produce flame-retardant paper to be used in the paper industry by using different paper coating pigments. In addition, it was investigated how much the produced paper coating affects the print quality.

2. MATERIALS AND METHODS

2.1 Materials and Methods

In the study, 80 g/m^2 high grade base paper was used. The papers were conditioned at 22°C and 55% humidity for 24 hours before the coating process. PCC, Zeolite and Bentonite were used as coating pigments. $CaCO_3$ (PCC) was supplied by Omya, Zeolite was supplied by ZO mineral partners and Bentonite was supplied by Imerys. The coatings were carried out automatically with a stick coater (model K303 Multi-coater, RK Print Coat Instruments Ltd, United Kingdom) on the base paper at a speed of 2 m/min. The coatings were allowed to dry for 24 hours at 20°C and 65% relative humidity (RH).

Test prints (Figure 1) on coatings were made with an inkjet Epson Stylus Photo R3000 digital printing machine and an X-Rite i1iO, X-Rite i1Pro Spectrophotometer and X-Rite eXact Portable Spectrophotometer were used for spectrophotometric and densitometric measurements to measure CIE $L^*a^*b^*$ values, color gamut volumes and dot-gain values on the printed paper. The measurement conditions of the spectrophotometer were a D50 light source in the range of 400-700nm and a polarizing filter with a geometry of 0/45° with a 2° observer angle. The difference between the colors of different prints was calculated according to the CIE ΔE_{00} color difference formula ISO 11664-6:2014. Chromix ColorThink Pro 3.0 was used to calculate the color gamut volumes.

$$\Delta E_{00} = \sqrt{\left(\frac{\Delta L'}{k_L S_L}\right)^2 + \left(\frac{\Delta C'}{k_C S_C}\right)^2 + \left(\frac{\Delta H'}{k_H S_H}\right)^2 + R_T \frac{\Delta C'}{k_C S_C}\frac{\Delta H'}{k_H S_H}} \quad (1)$$

where ΔL: is the lightness difference, ΔC: is the saturation difference, ΔH: is the hue difference, with correction using weighing coefficients (SL, SC, and SH) and constants called parametric coefficients (kL, kC, and kH) (El-Rashidy, Abdelraouf & Habib, 2022).

Thermogravimetric analyses (TGA) of coatings were performed using a Perkin–Elmer Thermogravimetric analyzer Pyris 1 TGA model. For color space measurements, a 400-piece i1Profiler Test Chart page (Figure 1) was used.

The flammability characteristics of composites were determined by Limited oxygen index (LOI). The LOI values of the coatings were measured by using an FTT (Fire Testing Technology) type instrument.

The crystalline phases of zeolite doped starch hybrid film, bentonite doped starch hybrid film and $CaCO_3$ (PCC) doped starch hybrid film were identified by X-Ray diffraction analysis (XRD, Rigaku Geigerflex D/Mac, C Series, Cu Kα radiation, Japan). Copper Kα radiation (λ = 1.5406 nm) produced at 30 kV and 25 mA scanned the range of diffraction angles (2θ) between 5° and 50° with a 2θ-step of 0.02°/s.

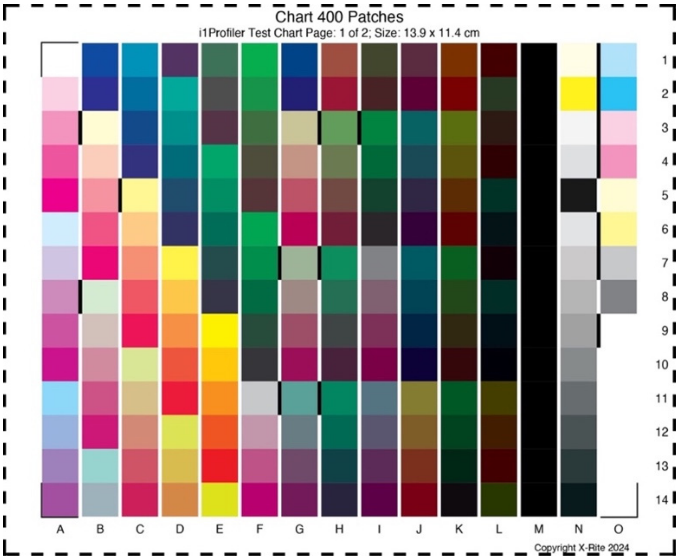

Figure 1: i1Profiler test chart page

3. RESULTS AND DISCUSSION

Paper formulations containing different pigments (PCC-starch, bentonite-starch and zeolite-starch) were successfully prepared and then coated on the base paper. Then, prepared test chart prints (Figure 1) on coatings were made with an inkjet Epson Stylus Photo R3000 digital printing machine.
The CIE *L*a*b** color values and dot gain values of the printed papers were measured with the X-Rite eXact Portable Spectrophotometer and the difference between the colors in the prints was calculated according to the CIE ΔE_{00} color difference formula ISO 11664-6:2014. The measurement results are given in the Table 1 and dot gain curves are given in the Figure 2. Chromix ColorThink Pro 3.0 was also used to calculate the color gamut volumes and the measurement results are given in the Table 2 and the 2D gamut volume curves are given in the Figure 3.

Table 1: Spectrophotometric color and color difference values of the papers used in the study

	ISO Lab Ref.			Base paper			ΔE_{00}
	*L**	*a**	*b**	*L**	*a**	*b**	
Cyan	55	-37	-50	61.6	-27.2	-39.8	7.27
Magenta	48	74	-3	54.1	65.3	-5.6	6.54
Yellow	91	-5	93	80.5	-9	78.9	7.91
Black	16	0	0	23.9	-3.6	-3.8	7.93
				PCC-Starch			
				57.8	-39.8	-48.1	2.94
				51.2	72.1	-3.8	3.25
				87.2	-6.2	89.8	2.58
				17.8	-2.1	-1.1	3.33
				Bentonite clay -Starch			
				57.8	-40.2	-49.6	3.16
				51.2	73.7	-5	3.3
				87.3	-6.9	87.4	2.84
				18.1	1.8	-2.1	3.47
				Zeolite -Starch			
				58.1	-39.1	-47.9	3.11
				50.6	72.2	-4.2	2.68
				86.9	-7.1	89.7	2.93
				18.2	-1.2	-0.8	2.4

Dot gain in printing is a feature that occurs in almost all printing and shows different variations in each. Dot gain is a measurable value that occurs between the printed dot value and the original dot value. As the dot sizes deviate from the standards, darker or lighter images are obtained in the print compared to the original, and this is not a desired result in printing.

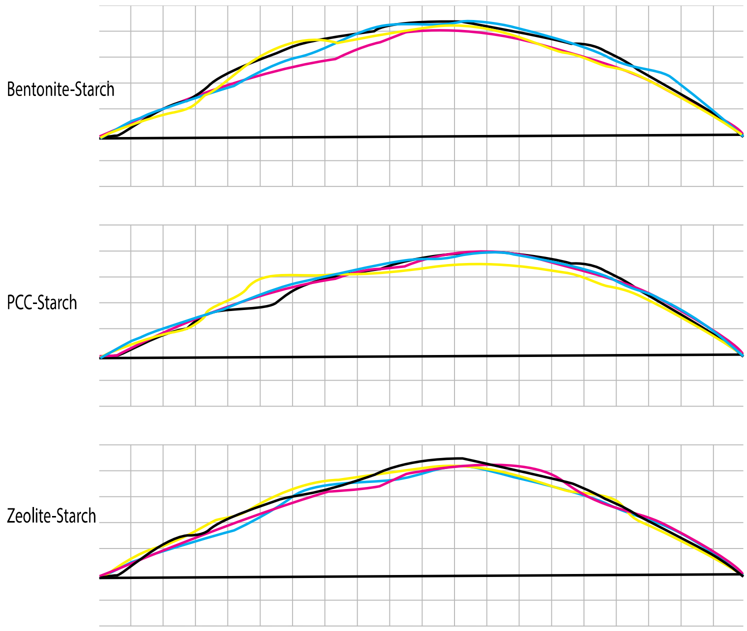

Figure 2: PCC-starch, bentonite-starch and zeolite-starch coatings dot gain curves

When the dot gain values for the bentonite-starch coating are examined, it is seen that the dot-gain value is at standard values for four colors in medium and dark tones, but there is a deviation in magenta color in light tones. For the PCC-starch coating, it is determined that the dot-gain is higher in yellow in light tones and lower in medium tones, and there is an irregularity in other colors. However, this irregularity is not seen in dark tones. For the zeolite-starch coating, it is determined that the dot gain value creates a more homogeneous, smooth dot-gain curve in all tones. The biggest reason for this is the difficulty of obtaining high quality prints using the heads of inkjet printers. These irregularities are also reflected in the irregularity of the dots obtained in the print (Shin, Fleming & Lee, 2021).

The CIE*L*a*b** color space is a three-dimensional, spherical model in which L* represents the brightness, *a** represents the red/green value, and *b** represents the yellow/blue value. *ΔE*ab* is defined as the difference between two colors in an *L*a*b** color space, where L represents the difference between each of the a and b readings compared to the initial readings.

When the uncoated base paper CIE*L*a*b** values were evaluated, it was determined that the color values measured in printing were significantly different from the reference values. According to the calculated ΔE_{00} values, it was seen that all ΔE_{00} values except magenta were outside of the acceptable values. PCC-starch coated papers show that CIEΔE_{00} color differences are below 3 in cyan and yellow values, indicating that color differences cannot be distinguished by eye. Magenta and black values are within acceptable values. The bentonite clay -starch coating values are examined; the yellow value is below 3 while the other 3 color values are within acceptable values. The zeolite-starch coating values were examined, it was determined that all color values except cyan were at values that could not be distinguished by the eye, but the cyan color value was very close to 3 and was within acceptable values.

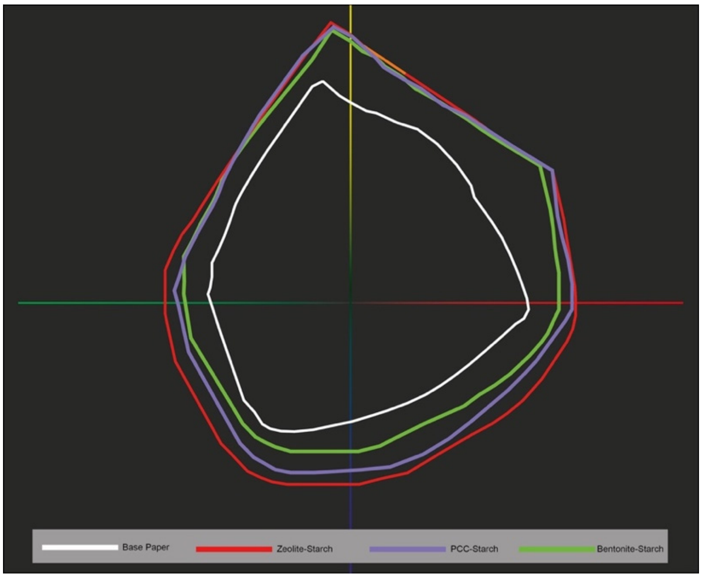

Figure 3: Base paper PCC-starch, bentonite-starch, and zeolite-starch coatings color gamut values

When all coatings and base paper 2D color gamut values are compared, it is seen that the base paper color gamut value creates a narrower area in all colors. The color gamut of matte paper is lower because uncoated papers give a lower density (D) because of their greater ink absorption and more microporous surface (Gigac, Stankovská & Pažitný, 2016), this resulting a loss of saturation, that is, the worst color gamut (Perales et al., 2009). When the coatings are compared with each other, it is seen that all coatings have the same area in yellow tones in the +b direction. Zeolite-starch has the widest green tones in the -a direction. PCC-starch coating and bentonite-starch coating values are very close to each other. In the +a direction, PCC-starch and zeolite-starch coatings are closer to each other in red tones and have a wide area. In the -b direction, the coating with the widest blue tones is zeolite-starch coating, followed by PCC-starch and bentonite-starch coatings. This can be explained by the fact that calcium carbonate pigments form coating layers with higher permeability and a more open pore structure, which allows rapid dye fixation. The use of starch, a cationic polymer, in the formulation is, as might be expected, beneficial to the cyan color (Hladnik, Cernic & Bukosek, 2008). Zeolites obtained from kaolin's efficiently adsorb methylene blue, safranin, and malachite green from aqueous solutions (Pereira et al., 2018). This explains why zeolite-starch coating creates a wider color gamut in both green and blue tones.

Table 2: Color gamut volume values

Coating Type	Volume
Base paper	211.779
PCC-starch	328.184
Bentonite-starch	298.517
Zeolite-starch	372.412

When the color volume gamut values obtained with Chromix ColorThink Pro 3.0 were compared, it was determined that the zeolite-starch coating had the widest color volume value (372.412). The narrowest color universe volume value (211.779) belonged to the uncoated base paper. The PCC-starch coating value (328.184) is quite wide compared to the bentonite-starch coating value (298.517).

Thermal behaviors of coated papers with different contents were investigated with TGA (Figure 4). When TGAs were examined, the decomposition curve of the base paper started at 320°C. It was determined that the thermal decomposition temperatures of the TGA curves increased slightly with the addition of fillers. This shows that the added fillers increased thermal stability. When the ash amounts of all coatings were examined, it was seen that they had more ash than the base paper, which can be attributed to the inorganic substances remaining without decomposition at 750°C. When the fillers were compared with each other, the fact that calcium carbonate and clay had very close curves and zeolite had a lower decomposition temperature is due to the decomposition of natural zeolite at 750°C and calcium carbonate and clay at

800°C. Addition of all relevant fillers to the medium changed the melt rheology in the coating and delayed the burning rate. The results are consistent with the literature (Md Nasir et al., 2019).
When the figure is examined, it is determined that the addition of zeolite, PCC and bentonite ignites the coating at a higher rate of oxygen, that is, delays combustion. It is also seen from the figure that zeolite provides more flame retardancy than clay and PCC. The reason for this is PCC and clay are hardly considered as an inorganic flame retardant. Still the decomposition of this inorganic materials is releasing some carbon dioxide. This is an endothermic process. Zeolite, which is an intumescent fire retardant, can form a protective barrier on polymer matrix surface and therefore increases charring. this is same line with TGA.

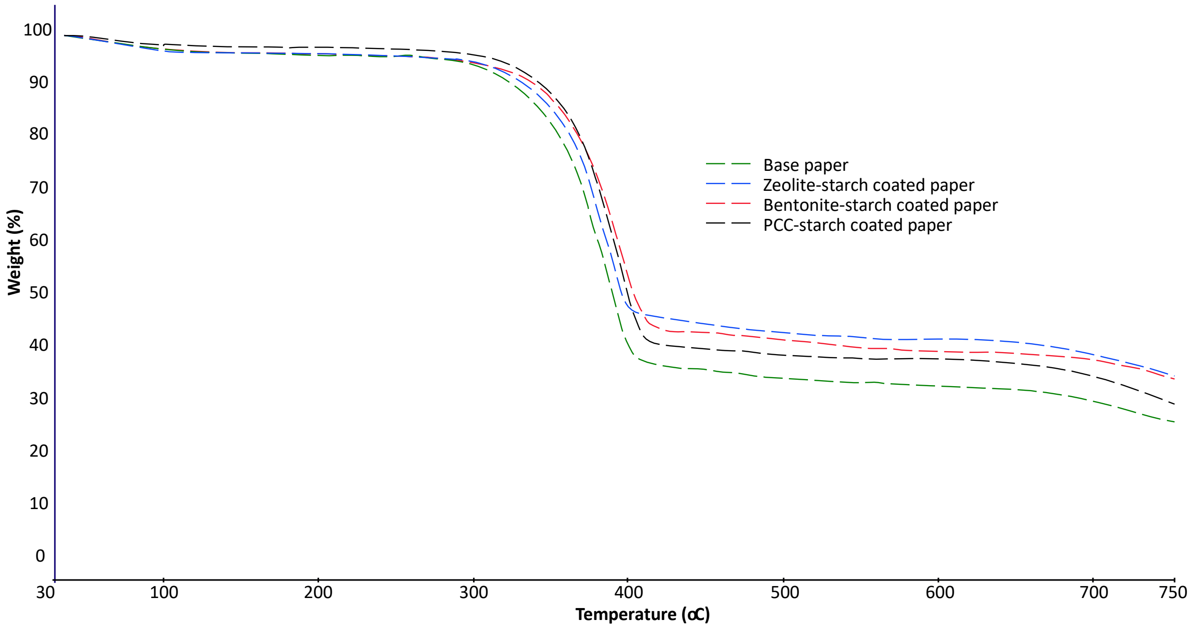

Figure 4: TGA thermograms of base paper PCC-starch, bentonite-starch, and zeolite-starch coatings

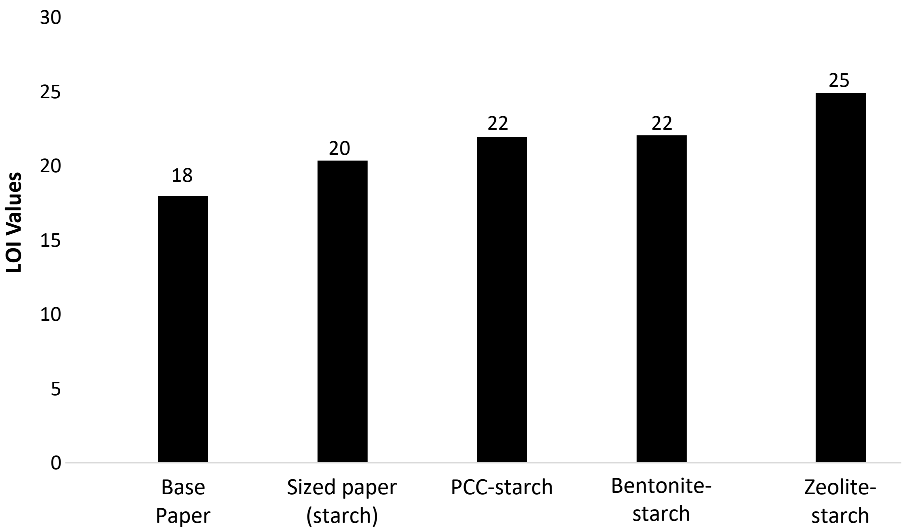

Figure 5: LOI values of base paper PCC-starch, bentonite-starch, and zeolite-starch coatings

Flame retardancy properties of paper coatings obtained with the limiting oxygen index technique (one of the most widely used techniques for determining flame retardancy) were examined. The measurement results are given in Figure 5. As can be understood from the figure, the LOI value of all coatings, especially zeolite, increased.

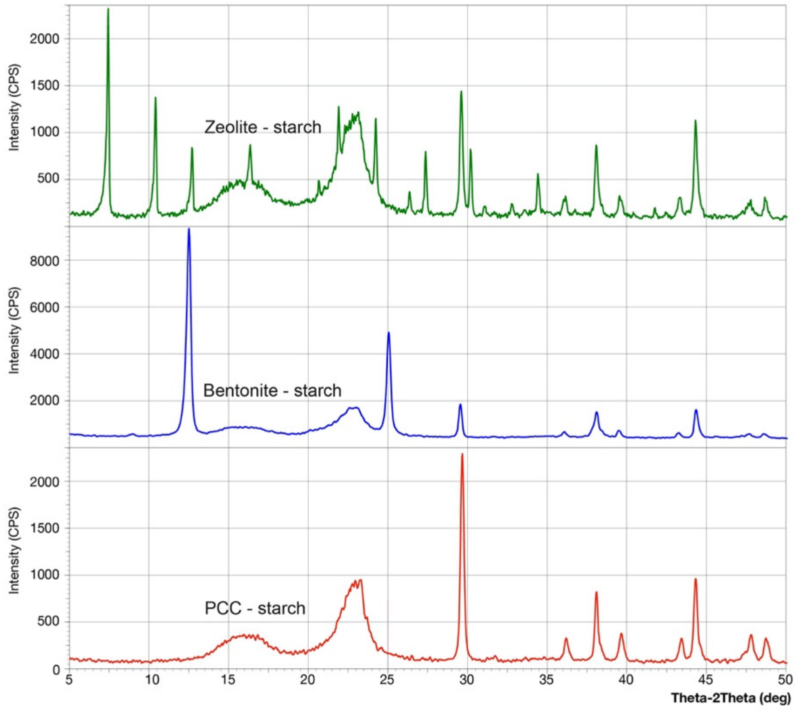

Figure 6: X-Ray diffractograms (XRD) of zeolite doped starch hybrid film (green), bentonite doped starch hybrid film (blue), $CaCO_3$ (PCC) doped starch hybrid film (red)

The X-ray diffraction patterns of zeolite doped starch hybrid film, bentonite starch doped hybrid film and PCC doped starch hybrid films are shown in Figure 6. When zeolite added starch film is examined, two peaks between 2θ = 20° and 2θ = 15° clearly show the semicrystalline structure of starch. In addition; the peaks occurring at 2θ = 7.37; 10,13; 30; 45° are the crystallinity peaks of the zeolite consistent with the literature (Belibi et al., 2013). When XRD bentonite doped starch spectrum is examined, three strongest peaks are observed which are 2θ = 12,46°, 25,01°, 29,49°. 2θ which may be assigned to aluminosilicate plates, corresponding to the basal spacing of kaolinite Si2Al2O5(OH)4 (d=7.09 Å) and shoulder (d=3.55 Å), respectively. These results are in line with the literature (Romo-Uribe et al., 2023). When the PCC doped starch film is examined, three peaks between 2θ = 24,11°, 36,14 ° and 39,58° are proves the crystalline structure of calcium carbonate and are supported by literature (Syafri, 2017). The simultaneous release of starch peaks clearly demonstrates the formation of the hybrid film.

4. CONCLUSIONS

In this study, PCC-starch, bentonite-starch, and zeolite-starch coatings were prepared and applied on high grade base paper. After the coating process, the coated papers showed much better properties than the base paper. Improvements in printing were detected in the base papers, which had a smoother surface after coating.

When the coatings were compared with each other, the zeolite-starch coating dot-gain values could be obtained homogeneously in all tones. This result is also clear when the color universe volumes are compared. When the 2-dimensional color universes are compared, the zeolite-starch coating has the widest color universe in the +a red area, -a green area and -b blue areas. It has a very high color gamut, especially in blue tones.

When the TGA curves of the coatings were examined, it was determined that all three different fillers triggered thermal decomposition at higher temperatures, that is, delayed the decomposition of the coating. The LOI results showed that all the coatings provided delayed ignition, and the best result was the coating containing zeolite. When these two results were examined, it showed that inorganic fillers were quite effective in cases where delayed ignition and non-flammability were sought.

XRD results show that all inorganic materials are crystalline without any chemical bonding and are fully compatible with the film. As a result, it has been seen that zeolite, PCC and bentonite are good additives in cases where late flame retardancy or non-flammability is sought.

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