

USE OF AGRICULTURAL WASTE IN PRINTING SUBSTRATES FOR GREEN PACKAGING IN MULTICOLOUR PRINTING

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Constant population growth and urbanization lead to increased waste generation, which contributes significantly to environmental degradation. Along with proper management of solid waste, an important guideline is to convert as much waste as possible into new functional products. In the graphic industry, the greatest innovations are in the use of more environmentally friendly raw materials, particularly in the field of packaging. The aim of this research was to analyse the effects of adding non-wood fibres in the production of screen-printed printing substrates on the degree of ink penetration into the substrate. From the comparison of all substrates, it can be concluded that the lowest ink penetration values into the printing substrate were achieved for all ink combinations with printing substrates containing 30% triticale pulp. The statistical analysis confirmed that the composition of the printing substrate has a significant influence on the penetration depth of the ink, while the influence of the different ink combinations is obvious, but limited.

Keywords: agricultural waste, mathematical models, penetration depth of the ink, printing substrate, screen printing

INTRODUCTION

One of the world's global problems is environmental pollution, which is caused exclusively by human activity, whereby the unbalanced exploitation of natural resources increases pollution.^{1,2} Rapid population growth and urbanisation increase waste production, which contributes significantly to environmental degradation. Any material that is no longer needed by the owner, producer or processor and no longer has any use is defined as waste.^{3,4} Therefore, waste needs to be differentiated according to its origin, so that it can be separated and further processed. There are many different types of waste, including municipal solid waste, agricultural waste, biomedical waste, industrial waste and hazardous waste. Municipal solid waste is waste generated by households or commercial activities, while agricultural waste includes livestock waste, agricultural crop residues and agro-industrial by-products.⁵ The amount of total waste is expected to increase to 3.40 billion tons per year by 2050. It is important to note that the largest generators, around a third of all waste, come from Asia, particularly China and India. Solid waste management is a major societal and political challenge, as the decomposition of waste causes

about 5% of greenhouse gas emissions that exacerbate climate change. Therefore, it is very important to be guided by the idea that every component of waste can be used to transform it into a functional new product.⁶

In the graphic industry, the biggest changes are in the use of more environmentally friendly raw materials, particularly in the field of packaging. Increasing emphasis is being placed on ensuring that the materials used to manufacture a particular graphic product fulfil not only their function, but also the option of sustainability and biodegradability.⁷ In order to utilize agricultural waste, several studies were conducted on the use of this type of waste in the processes of creating new, more environmentally friendly printing substrates.

The use of non-wood sources of cellulose fibres, including straw as agricultural waste, in the production of paper and board printing substrates is still insufficient and accounts about 8% of global paper production.⁸

In general, the sources of non-wood fibres used in papermaking can be divided into three categories depending on their origin: agricultural crop residues (*e.g.* sugarcane bagasse, corn stalks, cotton stalks, rice straw and wheat straw), naturally

growing plants (*e.g.* bamboo, esparto, reeds, sabai grass, papyrus, napier grass and invasive alien plants) and industrial crops (*e.g.* ramie, cotton fibre, kenaf, abaca and jute).⁹ In this study, agricultural waste, *i.e.* crop straw that is collected after annual harvesting and usually discarded or incinerated, was used as an additional fibre source for the production of printing substrate. Several studies have researched the use of agricultural waste to create paper or board, but very few analysed the printability of paper made from non-wood fibres.^{10,11}

In the actual production of a particular graphic product, the quality of the print depends not only on the printing speed, the printing pressure, printing substrate, and ink properties, such as viscosity, as well as temperature and the humidity in the printing house, but also on the depth of ink penetration into the printing substrate. The value of the ink penetration depth depends equally on the physical and chemical properties of the printing substrate defined by their composition and the properties of the ink used in printing process.¹² The drying of the ink on the printing substrate surface takes place in several stages, starting with primary drying on the surface of the print, followed by evaporation, absorption and diffusion. If the ink penetrates slowly into the substrate, the print dries more slowly, which can lead to a deterioration in the quality of print. On the other hand, if the ink has an extremely high penetration, the ink will diffuse between the cellulose fibres, resulting in a reduced ink layer on the surface of the print, *i.e.* the ink application will be insufficient.¹³

Knowing the importance of the interaction of the printing substrate and printing ink, the effects of the addition of non-woody lignocellulosic fibres obtained from wheat, barley and triticale stalks in the pulp for printing substrate production on the degree of ink penetration into the substrate is important to examine for the possibility of using this type of waste for the paper industry. In this research, printing will be performed using the screen printing process, in which the viscosity of the ink depends on the properties of the ink and the mesh. In the printing process, the ink is forced through a screen with a stencil using a doctor blade and transferred to the printing surface where drying occurs by evaporation of the solvent (water), and is accelerated by the supply of warm air. With this printing technique, it is possible to achieve an ink layer up to 12 μm thick on the print.¹⁴

Several methods are known for measuring the penetration depth of the printing ink into the printing substrate, which differ in their sensitivity, accuracy, sample size, sample preparation and thus in their suitability. They are usually roughly divided into non-destructive and destructive analyses. Destructive methods are based on microtome and microscopic analyses, such as scanning electron microscopy (SEM), secondary ion mass spectroscopy (SIMS), analyses with a focused ion beam (FIB) instrument or fluorescence recording confocal laser scanning microscopy (CLSM). The non-destructive methods for determining the penetration depth of the ink usually are based on the Kubelka-Munk theory.^{15,16,17}

Since the penetration depth of the printing ink is one of the factors that significantly influence the ink application and determine the final quality of the print, this study analyses the influence of the printing substrate with the addition of 30% crop straw on the penetration depth of the ink into the paper substrate in multicolour screen printing. To gain a deeper understanding of how different paper and ink combinations affect ink penetration, a statistical analysis based on microscopic measurements was conducted. Relative ink penetration depth was analysed using appropriate non-parametric methods due to non-normal data distribution. Kruskal-Wallis and Bonferroni post-hoc tests were performed to examine the interaction between paper substrate composition and ink combinations.

EXPERIMENTAL

Methodology

The experimental part was divided into the following steps: (1) the production of the pulp and printing substrates; (2) the printing of the printing substrates using screen printing technology; (3) the evaluation of the ink penetration depth; (4) the statistical analysis of the influence of the ink penetration depth as a function of the printing substrate.

Production of pulp and printing substrate

Straw as a fibrous raw material was collected from the field after the harvest of wheat (*Triticum* spp.), barley (*Hordeum vulgare* L.) and triticale (*Triticale* sp.) in the continental part of Croatia. The length of virgin fibres depended on the type of straw: for wheat straw, it was 0.83 ± 0.26 mm; for barley, 0.91 ± 0.29 mm; and for triticale, 0.93 ± 0.61 mm.¹⁸

After the purification process, the collected straw was first converted into a pulp using the soda pulping process, in which sodium hydroxide, heat and pressure

are used to dissolve the lignin that allows the fibres to bond together during the papermaking process by forming hydrogen bonds between their cellulose surfaces.¹⁹ The pulp was washed with tap water to remove all soluble pulp components and beaten in a Valley Hollander before being mixed with recycled wood pulp at a ratio of 3:7 in an Enrico Toniolo disintegrator. To ensure the uniformity and consistency of the pulp, a Frank PTI pulp homogenizer was also used after disintegration process.

In the final step, standardized paper sheets (\varnothing 200 mm) were produced in the laboratory from the prepared mixed pulp on an automatic papermaking machine, the Rapid-Köthen Automatic Sheet Former.

In this way, a sustainable alternative to traditional wood pulp paper was created, as the agricultural waste was used as a source of virgin fibres to reinforce the pulp made from recycled fibres in printing substrates production.²⁰ To determine whether this positive impact on the environment also has a positive impact on the print quality of these papers, a laboratory printing substrate consisting only of pulp made from recycled fibres (R) was produced and printed and further used as a reference sample during the research. A commercial printing substrate consisting only of pulp made from recycled fibres (K) was also printed and used as a control sample. Table 1 contains the abbreviations, and some properties of all printing substrates used in this research.

Printing of printing substrates using screen printing

The screen printing technique is mainly used today for printing detailed illustrations, special effects, self-adhesive labels, pharmaceutical products and outdoor applications and is often a unit integrated into an offset, flexographic and letterset machine. The quality of the print achieved with this printing technique is closely related to the material from which the screen is made, *i.e.* the fineness of the mesh (threads/cm or lin/cm), and the quality of the stencil. A wider range of inks is available for screen printing than for any other printing technique. With this technique it is possible to produce very high-quality prints with the fine line or tone with equal success.^{14,22}

For this research, all printing substrates were printed on a semi-automatic Shenzhen Juisun machine using a doctor blade with a mechanical hardness of 75 Shore and a mesh size of 120 lin/cm, at a temperature of 23 °C and a relative humidity of 50%. The printing process itself was carried out with Epta Hi-Gloss inks from KIIAN S.p.A.

In order to achieve a high print quality after printing, the prints were additionally dried in a tunnel device NP-2413 (380V) from HIX Corporation with warm air at a temperature of 55 °C. After printing, integral ink densities were achieved on the printing substrates and measured with a SpectroEye densitometer manufactured by X-rite under standard illumination D50, status E, and a 2° viewing angle (Table 2).

Table 1
Abbreviations and properties of printing substrates

Paper substrate	Thickness, T (μm) ¹⁹	Porosity (%)	Ash, w (%) ¹⁹	Roughness, R _a (μm) ^{19,21}
K	63.20 \pm 2.90	56.32 \pm 1.07	10.00 \pm 0.04	2.57 \pm 0.32
R	94.0 \pm 2.79	69.82 \pm 0.55	4.73 \pm 0.22	4.15 \pm 0.34
70R30W	101.5 \pm 5.32	70.99 \pm 1.15	3.64 \pm 0.07	4.59 \pm 0.51
70R30B	99.1 \pm 4.06	68.36 \pm 0.93	3.32 \pm 0.67	4.24 \pm 0.41
70R30T	99.4 \pm 6.20	69.47 \pm 0.47	3.99 \pm 0.15	4.40 \pm 0.39

Table 2
Integral ink density in screen printing prints

Paper substrate	Cyan	Magenta	Yellow	Black
K	1.469 \pm 0.009	1.210 \pm 0.023	0.822 \pm 0.008	1.502 \pm 0.023
R	1.305 \pm 0.020	1.127 \pm 0.014	0.806 \pm 0.008	1.314 \pm 0.018
70R30W	1.289 \pm 0.015	1.138 \pm 0.007	0.793 \pm 0.021	1.287 \pm 0.019
70R30B	1.287 \pm 0.013	1.075 \pm 0.017	0.767 \pm 0.013	1.253 \pm 0.021
70R30T	1.311 \pm 0.028	1.115 \pm 0.018	0.760 \pm 0.011	1.305 \pm 0.021

Evaluation of ink penetration depth

The penetration of printing ink into the printing substrate was determined based on microscopic observation of cross-sections of printed substrates. This method was chosen because it best describes the interaction between paper and printing ink. Prior to the microscopic analysis, the printed substrates with a size

of 10 mm \times 30 mm were poured with a mixture of 15:2 volume proportions of Epofix epoxy resin (with bisphenol diglycidyl ether) and Epofix hardener (with triethylene tetramine). After a drying period of 12 hours at room temperature without pressure, the samples were moulded and were ready for grinding and polishing. Grinding was carried out on a Buehler machine, and

polishing was performed on a Struers DAP-V machine with abrasive paper of different grit sizes and polishing pastes to obtain a smooth cross-sectional surface.

An Olympus GX 51 light microscope with 200x magnification and AnalySIS® software were used to observe the cross-sections of the printed printing substrates. The images obtained were additionally filtered and divided into 10 sections using ImageJ 1.54 software (bundled with Java 8).

Using this image analysis software, the ink penetration values of the observed cross-section (l) in micrometres and the local paper thickness (d), also in micrometres, were measured. The maximum ink penetration depth (Hp) or the relative ink penetration depth was calculated using the following equation:

$$Hp, \% = \frac{l}{d} \times 100 \quad (1)$$

Selection of statistical tests

Prior to inferential analysis, the normality of ink penetration data was assessed using the Shapiro–Wilk test ($p = 0.5071$), indicating no significant deviation from normal distribution. However, to ensure methodological consistency across all analyzed variables and to minimize the influence of potential outliers, non-parametric methods were applied throughout the study. The Kruskal–Wallis test was used to evaluate differences among paper substrates, followed by Bonferroni-adjusted post hoc comparisons when significant differences were detected. The interpretation of the results additionally considered

relevant substrate properties, including thickness, ash content, and surface roughness, due to their potential influence on ink penetration behavior.

RESULTS AND DISCUSSION

The penetration of the printing ink into the printing substrate was observed by microscopic cross-sectional images of the multicolour printed printing substrates. Figure 1 shows microscopic cross-sectional images of all printing substrates (K, R, 70R30W, 70R30B, 70R30T) printed with magenta ink over previously printed yellow to produce a red colour. In microscopic images, the penetration of the ink into the substrate was defined in percentages using the ImageJ program.

The microscopic cross-sectional images of the prints clearly show cellulose fibres and printing ink, with the printing ink penetrating more strongly into the printing substrate in the printed samples R (Fig. 1b) and 70R30B (Fig. 1d).

Based on the microscopic observation of the cross-section of the printed printing substrate, the penetration of the printing ink into the printing substrate was determined on 10 sections in each image, and the calculated average value of the ink penetration into each printing substrate is shown in percentages (relative ink penetration) in Figure 2.

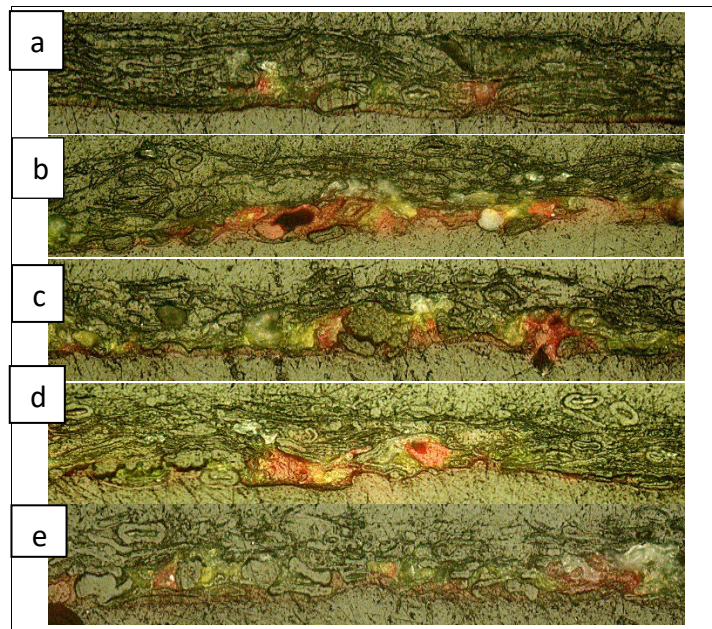


Figure 1: Microscopic cross-sectional images of red prints achieved with Y+M inks taken under 200x magnification on substrates a) K; b) R; c) 70R30W; d) 70R30B; e) 70R30T

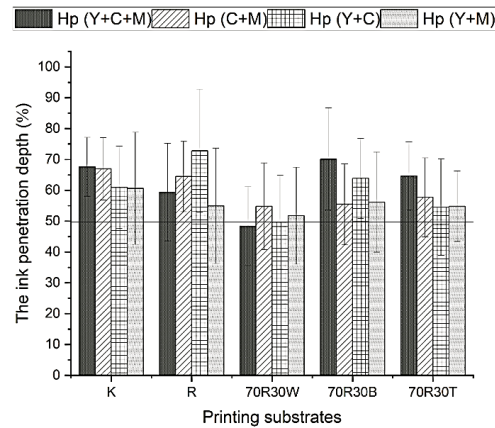


Figure 2: Relative ink penetration, Hp – average value of ink penetration into the printing substrate in percentages

Observing the results of relative ink penetration within the printing substrate (Fig. 2), it can be seen that only the print obtained with two inks Y+C shows an increased penetration on the R substrate ($H_{pR} = 72.78\%$), while most of the other prints presented the highest ink penetration when printed with three colours Y+C+M ($H_{p70R30B} = 72.78\%$; $H_{pK} = 67.58\%$; $H_{p70R30T} = 64.63\%$). Prints on the 70R30W substrate have the lowest penetration values, which are the same regardless of the ink combination used ($H_{p70R30W} = 48.40\% - 54.79\%$). All prints obtained with two inks Y+M have the lowest ink penetration values regardless of the type of printing substrate.

To statistically evaluate the effect of ink application on penetration (Hp) across different printing substrates, the influence of substrate composition and ink combination on ink distribution was analysed, expressed as a percentage of the total paper thickness.

Influence of ink application on the penetration of ink into paper substrate

The influence of ink application on penetration, expressed as a percentage of the total paper thickness, was evaluated. For this purpose, the Kruskal–Wallis test was applied separately for each paper substrate (Tables 5 and 6).

The percentage of penetration, with respect to the total paper thickness ($T = 101.67 \mu\text{m}$), remains consistent and this consistency suggests a balanced absorption structure in 70R30W paper, where capillarity and porosity allow for proportional ink distribution throughout the material, regardless of ink type. The addition of 30% virgin wheat fibres to recycled pulp improves the predictability of ink

penetration characteristics, contributing to more uniform printing results.

Despite some variations in penetration depth values, the percentage of ink absorption remains consistent in the 70R30B print, indicating a uniform absorption profile throughout the paper thickness ($T = 91.67 \mu\text{m}$).

Ink application has no statistically significant effect on ink penetration in 70R30T paper, as shown by the Kruskal–Wallis test ($p = 0.3618$). The distribution around the overall median (56.24%) shows a balanced spread across all ink combinations (Y+M, Y+C, C+M, Y+C+M), with no notable group differences. The consistent penetration profile throughout the paper's thickness ($T = 101.67 \mu\text{m}$) indicates that 70R30T paper has a uniform absorption structure supporting even ink distribution. Physical characteristics, such as low ash content ($w = 3.99\%$), enhancing capillary flow, and moderate surface roughness ($R_a = 4.40 \mu\text{m}$), enabling controlled surface retention, contribute to the consistent ink behaviour. These combined properties make 70R30T paper suitable for printing applications requiring consistent and predictable absorption.

Post-hoc analysis

Since ANOVA could not be applied due to the characteristics of the dataset, the Kruskal–Wallis test was used as the main method for comparing penetration between paper substrates. As this test only indicates the presence of statistically significant differences among groups, a post-hoc analysis was conducted to identify which specific groups differed. For this purpose, the Bonferroni test was applied as a conservative approach that

controls the risk of Type I error in multiple comparisons. This analysis addresses the following questions:

- Which paper substrates differ significantly in penetration?
- Are there groups with similar penetration patterns?

The Bonferroni post-hoc test was applied to examine in more detail the differences in

penetration between the paper substrates, and the results are presented in Table 7. The Bonferroni post-hoc test was applied to ranked data obtained from the Kruskal-Wallis analysis to determine statistically significant pairwise differences in relative ink penetration between paper substrates.

Table 5
Results of the Kruskal-Wallis test for ink penetration in all paper substrates

Paper substrate	Dependent: ink penetration
K	Median Test, Overall Median = 63.4742; Ink Penetration (Paper K) Independent (grouping) variable: Ink Application Chi-Square = 1.600000 df = 3 p = 0.6594
R	Median Test, Overall Median = 63.3568; Ink Penetration (Paper R) Independent (grouping) variable: Ink Application Chi-Square = 2.400000 df = 3 p = 0.4936
70R30W	Median Test, Overall Median = 51.4806; Ink Penetration (Paper 70R30W) Independent (grouping) variable: Ink Application Chi-Square = 4.000000 df = 3 p = 0.2615
70R30B	Median Test, Overall Median = 61.2385; Ink Penetration (Paper 70R30B) Independent (grouping) variable: Ink Application Chi-Square = 3.200000 df = 3 p = 0.3617
70R30T	Median Test, Overall Median = 56.2429; Ink Penetration (Paper 70R30T) Independent (grouping) variable: Ink Application Chi-Square = 3.200000 df = 3 p = 0.3618

Table 6
Kruskal-Wallis median test results for ink penetration by paper substrate and ink application

Dependent: ink penetration	Median Test, Overall Median = 63.4742; Ink Penetration (Paper K) Independent (grouping) variable: Ink Application Chi-Square = 1.600000 df = 3 p = 0.6594				
	Y+M	Y+C	C+M	Y+C+M	Total
<= Median: observed	6.00000	6.00000	4.00000	4.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	1.00000	1.00000	-1.00000	-1.00000	
> Median: observed	4.00000	4.00000	6.00000	6.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	-1.00000	-1.00000	1.00000	1.00000	
Total: observed	10.00000	10.00000	10.00000	10.00000	40.00000

Dependent: ink penetration	Median Test, Overall Median = 63.3568; Ink Penetration (Paper R) Independent (grouping) variable: Ink Application Chi-Square = 2.400000 df = 3 p = 0.4936				
	Y+M	Y+C	C+M	Y+C+M	Total
<= Median: observed	5.00000	4.00000	4.00000	7.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	0.00000	-1.00000	-1.00000	2.00000	
> Median: observed	5.00000	6.00000	6.00000	3.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	0.00000	1.00000	1.00000	-2.00000	
Total: observed	10.00000	10.00000	10.00000	10.00000	40.00000

Dependent: ink penetration	Median Test, Overall Median = 51.4806; Ink Penetration (Paper 70R30W) Independent (grouping) variable: Ink Application Chi-Square = 4.000000 df = 3 p = 0.2615				
	Y+M	Y+C	C+M	Y+C+M	Total
<= Median: observed	4.00000	6.00000	3.00000	7.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	

obs.-exp.	-1.00000	1.00000	-2.00000	2.00000	
> Median: observed	6.00000	4.00000	7.00000	3.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	1.00000	-1.00000	2.00000	-2.00000	
Total: observed	10.00000	10.00000	10.00000	10.00000	40.00000

Dependent: ink penetration	Median Test, Overall Median = 61.2385; Ink Penetration (Paper 70R30B) Independent (grouping) variable: Ink Application Chi-Square = 3.200000 df = 3 p = 0.3618				
	Y+M	Y+C	C+M	Y+C+M	Total
<= Median: observed	5.00000	5.00000	7.00000	3.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	0.00000	0.00000	2.00000	-2.00000	
> Median: observed	5.00000	5.00000	3.00000	7.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	0.00000	0.00000	-2.00000	2.00000	
Total: observed	10.00000	10.00000	10.00000	10.00000	40.00000

Dependent: ink penetration	Median Test, Overall Median = 56.2429; Ink Penetration (Paper 70R30T) Independent (grouping) variable: Ink Application Chi-Square = 3.200000 df = 3 p = 0.3618				
	Y+M	Y+C	C+M	Y+C+M	Total
<= Median: observed	5.00000	7.00000	5.00000	3.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	0.00000	2.00000	0.00000	-2.00000	
> Median: observed	5.00000	3.00000	5.00000	7.00000	20.00000
expected	5.00000	5.00000	5.00000	5.00000	
obs.-exp.	0.00000	-2.00000	0.00000	2.00000	
Total: observed	10.00000	10.00000	10.00000	10.00000	40.00000

Table 5

Results of the Bonferroni post-hoc test on ranked data for ink penetration between paper substrates

Paper substrate	Mean ink penetration (%)	K	R	70R30W	70R30B	70R30T
K	64.028	-	1.000000	0.001188	1.000000	0.635917
R	62.902	1.000000	-	0.004259	1.000000	1.000000
70R30W	51.146	0.001188	0.004259	-	0.020427	0.404157
70R30B	61.396	1.000000	1.000000	0.020427	-	1.000000
70R30T	57.911	0.635917	1.000000	0.404157	1.000000	-

Bonferroni test; variable depth % (experimental data set) probabilities for post-hoc tests error: between MSE = 214.94, df = 192.00

The results indicate that paper substrate K differs significantly in penetration from substrate 70R30W ($p = 0.001188$), while no statistically significant difference was observed between substrate K and other paper substrates. This suggests that the composition of all analysed paper substrates, with the exception of 70R30W, enables a similar distribution of ink throughout the paper thickness, reflecting comparable structural properties that influence capillary uptake.

Statistically significant differences for ink penetration between paper substrates are a consequence of internal structure of randomly laid fibres that vary in length and quality. The largest

difference was found between laboratory made substrate 70R30W and substrates without agricultural waste addition, K ($p = 0.001188$) and R ($p = 0.004259$), confirming that these substrates exhibit markedly different ink absorption characteristics.

Overall, 70R30B aligns more closely with paper substrates made without addition of straw cellulose fibres. On the other hand, paper 70R30W is statistically distinct from most other samples, likely due to its specific fibre structure and chemical composition affecting the ratio of ink absorption to total thickness.

These findings reinforce the importance of aligning printing parameters and ink formulations with the physical characteristics of each paper substrate to ensure consistency in print quality.

CONCLUSION

The results of this research indicate that the lowest ink penetration values for all analysed substrates were achieved when the samples were printed with the Y+M ink combination. The 70R30W substrate has the lowest ink penetration value compared to the other printing substrates. Comparing all substrates, it can be concluded that, for most substrates, the highest ink penetration values were obtained when printing with all three inks layered on each other (Y+C+M).

Since ink penetration was measured only in areas with maximum ink penetration within the printing substrate, the maximum ink penetration did not differ significantly between substrates. The length of straw cellulose fibres and the porosity of substrates did not significantly affect the ink penetration value.

Although minor differences between ink combinations (Y+M, Y+C, C+M, and Y+C+M) were noticed based on microscopic observation of cross-sections of printed substrates, statistical analysis (Kruskal–Wallis, $p > 0.05$) confirms a consistent ink penetration depth between ink combinations within the same substrate.

The results of the Bonferroni post-hoc test point out that the paper with the addition of 30% wheat straw is statistically distinct from other substrates with agriculture waste. Similar behaviour of ink penetration depth was achieved on paper substrates made without and with addition of barley and triticale straw.

This study provides scientific insight into the variability and consistency of the behaviour of multicolour printed paper substrates with the addition of agricultural waste.

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