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Heat-Induced Method for the Development of Sustainable Edible Paper Eco–Conscious Packaging

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The increasing environmental concerns related to pollution from traditional paper and plastic food packaging have spurred a new market trend towards developing eco-friendly alternatives, notably edible food packaging. This study focused on creating food packaging material using edible paper sourced from plants {Corn starch (CS), Rice starch (RS), Pectin (P), Carboxymethyl Cellulose (CMC), Xanthan Gum (XG), and Glycerol (G)}. The research delved into analyzing the structural and mechanical properties of the developed edible paper, utilizing SEM, XRD, tensile strength, elongation at break, and Young's modulus as key evaluation parameters. Among the various compositions tested, the HT11 {CS (15):RS(10):P(4)} blend displayed a significant tensile strength of 39.413 MPa, indicating its ability to withstand mechanical stress effectively. Additionally, the material exhibited remarkable flexibility, with an elongation at break value of 91.765 MPa. The range of Young's modulus values highlighted the material's adaptable stiffness and elasticity levels. Moreover, the material's amorphous nature and uniformity in XRD and SEM testing further emphasized its quality. Although FTIR and UV tests yielded negative results, these findings signify the ongoing advancements in edible paper technology, promoting sustainability, innovation, and culinary versatility in food packaging practices.

Keywords: Edible paper, Biodegradable, SEM, XRD, Mechanical properties.

1. Introduction

Food packaging plays a crucial role in maintaining the safety and quality of food products Vasile and Baican (2021). It protects food from contamination, extends shelf life, and allows for communication Alamri et al.,(2021). Biodegradable food packaging materials nowadays are gaining more importance due to their eco-friendly nature, and growing awareness of environmental issues related to the traditional use of packaging material Motelica et al.,(2020).

In the current era, the research and commercialization of biodegradable food packaging materials have emerged as alternatives to synthetic polymers and paper-based food packaging materials. These biodegradable materials offer eco-friendly and sustainable solutions, addressing environmental concerns associated with traditional packaging materials Ncube et al.,(2020). The shift towards biodegradable packaging materials is driven by consumer demand for environmental friendly products and the need to reduce the waste and carbon footprints in the food industry Ketelsen et al.,(2020).

Newspapers and printed pamphlets are commonly used as food packaging materials either as primary packaging material or secondary packaging material Ramakanth et al.,(2021). They are often used for wrapping, packing, and serving food, particularly in street food vendors and small-scale food businesses (hotels) Mwove et al.,(2020). They are also used for take-away and delivering segments of the food industry, where sturdy packaging is required for the transportation of food. These printed pamphlets are sometimes used to remove excess oil from deep-fried food An, (2022); Jadhav et al.,(2021) (Figure 1).

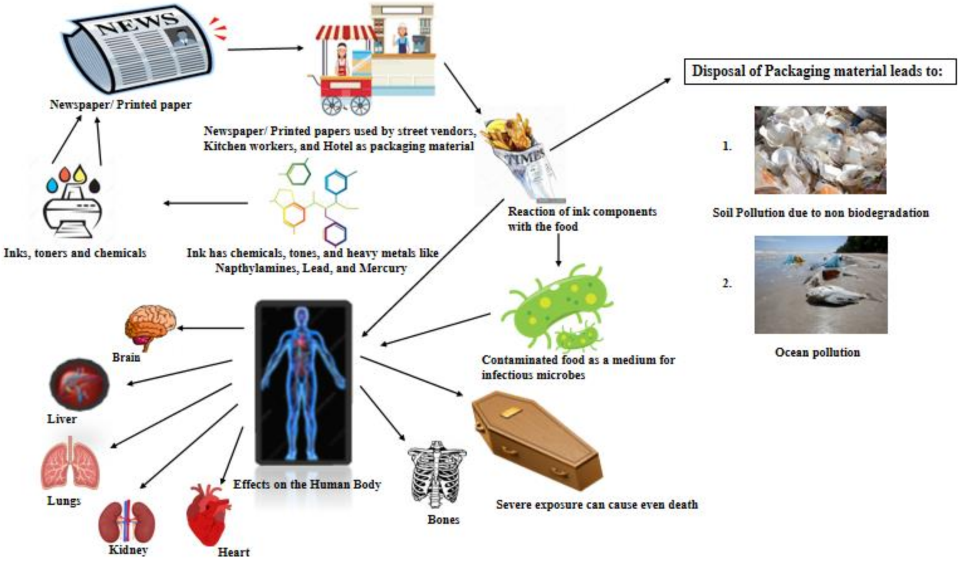


Fig 1. Health hazards of using newspaper/ printed paper as food packaging material

The ink used in printed paper can have adverse effects on food materials due to the presence of harmful chemicals and contaminants leading to various health risks and safety concerns (Fig. 1) Jadhav et al.,(2021). The newspaper contains approximately 3000mg / Kg of mineral oil which falls under a specific classification Biedermann and Grob, (2010). The Joint FAO/ WHO Expert Committee on Food Additives (JECFA) has established an acceptable daily intake of 0.01mg /kg body weight for these mineral oils Grob, (2018). The presence of mineral oils in newspapers used for food packaging raises concern about potential health risks associated with contamination and highlights the importance of adhering to safety standards to protect consumer wellness Coles, (2013).

Recently, the concept of edible paper has grabbed the attention of researchers which led to many innovative developments in the field of food packaging Firouz et al.,(2021). The use of starch-pectin and other natural-source dopants has been investigated, building on prior research, to develop new biodegradable food packaging materials. The choice and standardization of the starch sources and dopants were guided by experimental outcomes. The standardized materials underwent thermal processing, during which their mechanical, physical, and structural characteristics were assessed.

The study aims to enhance the eco-friendly, edible food packaging material through the use of natural-based compounds like starch, and pectin promoting a more sustainable approach to packaging solutions.

2. Materials and Methodology

The raw materials such as starch sources, Carboxymethyl cellulose, pectin, xanthan gum, and glycerol of food grade were obtained from the Institute of Baking and Cake Art, Bangalore. The experiment was conducted at the Department of Food Technology, Ramaiah University of Applied Sciences, Bangalore, India.

Preliminary Trials

Preliminary studies were conducted on the development of edible paper using various starches like tapioca, corn, and potato starch. The experiments revealed challenges such as uneven film surfaces, cluster formation, and high-degree cracks due to inadequate starch concentration. Despite adjustments in starch concentration, issues like film shrinkage and poor cross-linking persisted. Additional studies focused on the effects of pectin, glycerol, carboxymethyl cellulose (CMC), and xanthan gum on the paper's properties. Optimal outcomes were observed with specific concentrations of these additives, highlighting the importance of proper cross-linking for film formation. The inclusion of rice flour was explored to address stickiness, leading to further investigations into suitable concentrations for improved paper quality.

Preparation of casting solution

Following the initial investigation, the composition of the edible paper blend and the range of concentration levels were established. Utilizing the Box-Behnken approach within the framework of response surface methodology, Table 1 outlines the different concentration levels of parameters as determined by the Box-Behnken design.

Food-grade carboxymethyl cellulose and xanthan gum were individually dissolved in distilled water at 70°C until fully dissolved, then filtered to achieve a clear solution. This solution was subsequently mixed with corn starch, rice starch, and pectin to form a stable emulsion. Glycerol was incorporated to enhance the flexibility of the mixture. The resulting mixture was then poured into petri dishes and dried in a hot air oven at 65°C for 24 hours. Figure 2 illustrates the preparation process of the emulsion and its subsequent drying (Figure 2) (Table 1).

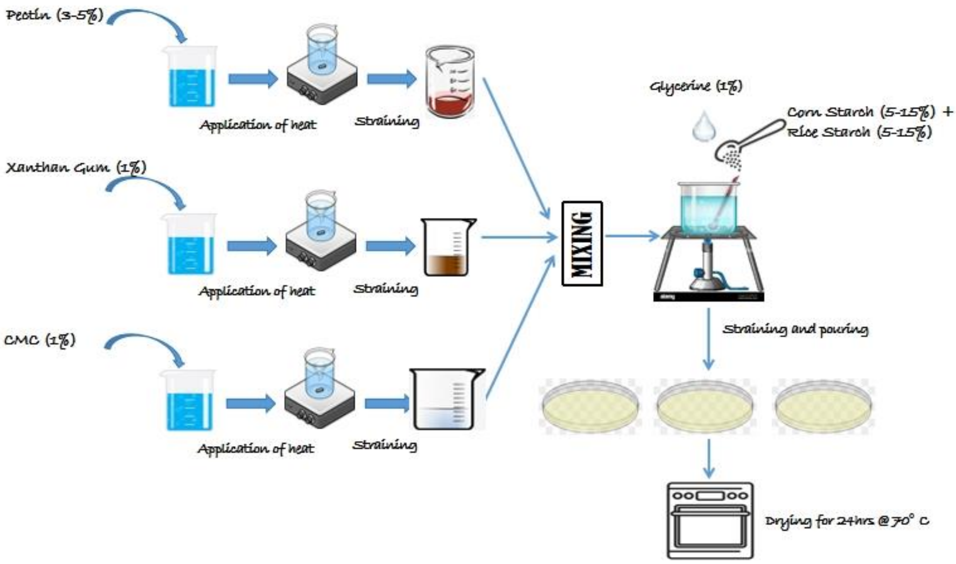


Fig. 2: Flow diagram for the preparation of starch edible paper

Table 1: Composition table of edible paper blends (w/v)

Sample Name	Corn Starch (%)	Rice Starch (%)	Pectin (%)
HT1	5	5	4
HT2	5	7.5	5
HT3	5	10	4
HT4	5	7.5	3
HT5	10	5	3
HT6	10	5	5
HT7	10	7.5	4
HT8	10	10	5
HT9	10	10	3
HT10	15	7.5	5
HT11	15	7.5	3
HT12	15	10	4
HT13	15	5	4

(Based on the preliminary studies, carboxymethyl cellulose (CMC), xanthan gum, and glycerol were maintained at a constant concentration of 1% throughout the subsequent experiments)

Film Characterization

Film thickness:

A high-accuracy Mitutoyo digital micrometer with a precision of 0.001mm was utilized to measure the thickness of the prepared active films at five distinct points, and the average thickness was subsequently calculated Hanumantharaju et al., (2019).

Moisture content:

The weight loss of the film might be measured using the ASTM D4442 method to determine the moisture content. The film samples were prepared into 2X2 cm square shape and precise weight measurements were made of the prepared specimen samples (W1). Subsequently, the specimen samples were dried at 100 °C for 24 hrs in an oven to achieve a consistent weight; the specimen's final weight was noted (W2). Three iterations of this process were carried out, and mean values were computed. Using the following formula, the films' moisture content was determined Shanbhag et al.,(2023).

$$\text{Moisture content } (\%) = \frac{(W_1 - W_2)}{W_2} \text{ X } 100$$

Where, the film's initial weight is W_(1), while its final weight is W_2.

Water solubility:

The water solubility of the prepared active films was assessed according to ASTM standard D570–98. Initially, film samples were pre-dried in hot air at 80°C for 24 hours, and their initial weight (W1) was recorded. Subsequently, the films were immersed in 30 ml of double-distilled water at room temperature for 24 hours, followed by squeezing out excess water using sanitary paper. Afterward, the films were dried again for a day at 100°C, and the final film weight (W2) was recorded Eelager et al.,(2023); Hanumantharaju et al., (2020) . The percentage of water solubility was calculated using the equation provided in the study.

$$\text{Water solubility } (\%) = \frac{(W_1 - W_2)}{W_2} \text{ X } 100$$

Where, the specimen samples' initial and final weights are denoted by W1 and W2.

Mechanical properties:

Mechanical properties of rectangular film samples were examined using a Universal Testing Machine (DAK, System Inc, Mumbai) Jemima et al., (2021). The samples measured 2.5 x 10 cm and were securely positioned within the extension grips of the machine. The properties examined included thickness, tensile strength (Ts), Young's modulus (Ym), peak load, and elongation at break (Eb). The stretching of the samples was conducted at a crosshead speed of 5 mm/min. The experiments were conducted at room temperature Narasagoudr et al.,(2020).

X-ray Diffraction (XRD):

Through X-ray diffraction analysis, the crystallinity of the blend films and its impact on their physical characteristics were determined. With a scanning speed of 2° min-1, the radiation was produced by a Cu Kα source operating at 30 kV and 20 mA in the $2\theta = 0°–40°$ range. Equipped with the diffracted intensity data, crystallinity was estimated Kasai et al.,(2018).

Scanning Electron Microscopy (SEM):

The morphological changes resulting from starch and other dopant blends were analyzed using SEM imaging with a JEOL JSM-6360 instrument operating at an acceleration voltage of 10 kV. To counteract the high electron beam's charging effect, all specimens were coated with a conductive layer of gold through sputter coating. Additionally, the film specimens were attached to metal stubs with double-sided adhesive carbon tape Narasagoudr et al.,(2020); Eelager et al.,(2023).

Statistical analysis

The Box Behnken design, a part of the response surface methodology, was chosen for the statistical optimization of the edible paper emulsion due to its suitability for experiments involving three factors at three levels. The variables selected for this study were corn starch (ranging from 5% to 15%), rice starch (ranging from 10% to 15%), and pectin (ranging from 3% to 5%), which are the primary components for preparing the edible paper emulsion. This design framework included a total of 13 experiments, which are detailed in Table 1.

3. Result and Discussion

Thickness

The thickness measurements of the 13 samples range from 0.05 mm to 0.27 mm (Table 2), playing a critical role in the durability, protection, and quality of food packaging materials. The thickness of the film has significantly decreased ($P < 0.01$) with an increase in the concentration of pectin from 3-5%, which can be attributed to the plasticizer property of pectin that forms a thin and continuous film. Notably, HT01 exhibits the highest thickness at 0.27 mm, while HT08 has the lowest at 0.05 mm, showcasing significant variability that can impact the strength and protective properties of the packaging material (Fig. 3). Edible films, on the other hand, are typically thin sheets with thicknesses ranging from 0.05 mm to 0.25 mm, serving as wraps for food products Hamed et al.,(2022). This consistency within the standard thickness range ensures that all samples meet the expected criteria for food packaging materials (Table 2) (Figure 3).

Table 2: Thickness of edible paper

Sl. No.	Thickness (mm)
HT01	0.27 ±0.04
HT02	0.14 ± 0.01
HT03	0.09 ± 0.01
HT04	0.22 ± 0.03
HT05	0.16 ± 0.02
HT06	0.15 ± 0.01
HT07	0.09 ± 0.01
HT08	0.05 ± 0.03
HT09	0.25 ± 0.06
HT10	0.12 ± 0.03
HT11	0.20 ± 0.01
HT12	0.19 ± 0.01
HT13	0.20 ± 0.01

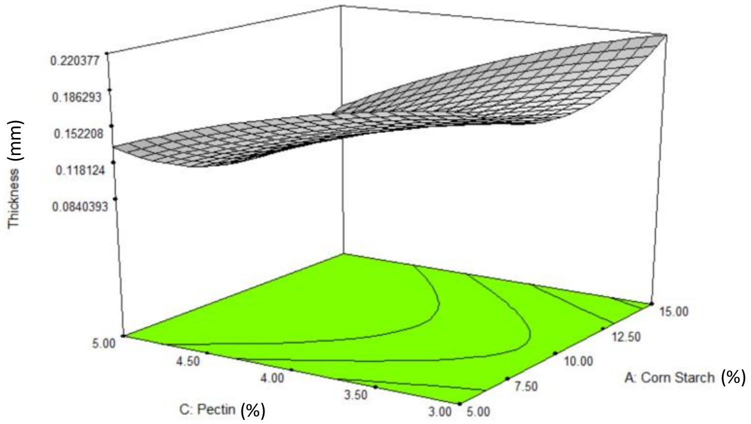


Fig. 3: Thickness of edible paper at different concertation of corn starch , rice starch and pectin

Moisture Content

Moisture content of packaging material contributes to the growth of microorganism on the surface when exposed to food and environment. Using corn starch and pectin together along with other dopants to produce the edible paper resulted in variation in the moisture absorption rate. Moisture content of the packaging varied between 8.45% and 23.75%. The observed decline in moisture content with the incremental concentration of corn starch from 5-15% was not statistically significant ($P<0.05$) (Fig. 4). This phenomenon could be attributed to the intrinsic water-binding capacity of corn starch, which tends to absorb and retain moisture within its granular structure. As the proportion of corn starch increases, it may absorb more water, leading to a lower moisture content in the overall material. However, the lack of statistical significance suggests that the variations in moisture content are within the range of natural variability and do not conclusively indicate a trend. Factors such as the uniformity of starch distribution, the granularity and specific properties of the starch used, and the precision of the moisture content measurement could all contribute to this outcome.

Sample HT 8 {CS(10); RS(10): P(5)} was thought to have the highest moisture content, and sample HT 3 {CS(5): RS(10): P(4)} has the lowest moisture content, this suggests that the water content of the edible paper was found to be fluctuating concerning change in the concentration of starch and pectin Dash et al.,(2019).

High water content adversely affects the dimensional stability of composite materials by compromising their mechanical strength, increasing porosity, and enhancing water-holding capacity as highlighted by Ibrahim et al.,(2020). Therefore, maintaining low moisture content is crucial to preserve the structural integrity and performance of composite materials, ensuring optimal mechanical properties and durability.

The analysis revealed that the typical moisture content of the edible film is approximately 8.3% Fransiska et al.,(2020). Notably, sample HT 11 exhibits a comparable moisture content level, and demonstrates favorable properties, aligning well with the standard moisture content for edible paper (Figure 4).

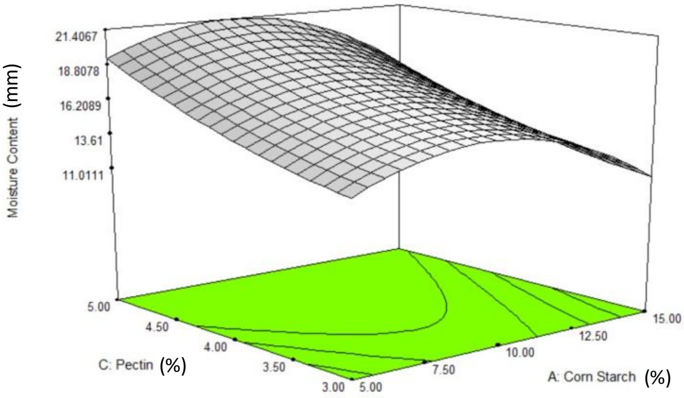


Fig. 4: Moisture content of edible paper at different concertation of corn starch, rice starch and pectin

Water solubility:

The water solubility of a film reflects its biodegradability and utility in food packaging applications. A critical factor in food packaging materials is the generated edible paper's solubility rate, which is influenced by both hydrophilic and hydrophobic constituents Petkoska et al.,(2021). The reduction in film solubility with an increase in corn starch concentration, although not statistically significant ($P<0.05$) (Fig. 5), can be attributed to the inherent characteristics of starch. Starch molecules, particularly amylose, have a tendency to form dense, less soluble matrices as their concentration increases, which could potentially reduce the overall solubility of the film. However, the minimal impact observed suggests that other factors, such as the interaction between starch and pectin or the presence of glycerol and other dopants, might also influence the solubility dynamics, balancing out the effect of increased starch concentration. The average solubility of the edible paper being 50% indicates a moderate interaction between the components, suggesting that while starch contributes to decreased solubility, the formulation's overall design maintains a certain level of solubility essential for its intended application.

HT 8 {CS(10): RS(10): P(5)} demonstrated the highest water solubility among all samples, while HT 11 {CS (15): RS(7.5): P(3)} exhibited the lowest water solubility. According to Patil et al. (2021), increased biodegradability is linked to higher solubility due to the superior interactions hydrophilic materials have with soil and water. Notably, starch-based edible films typically have water solubility rates ranging from 23% to 35%, placing sample HT 11 within this range Shanbhag et al., (2023). This suggests that a higher solubility rate facilitates the biodegradation process by making the material more accessible to microbial action, ultimately promoting the conversion of the susbstance into simpler organic components (Figure 5).

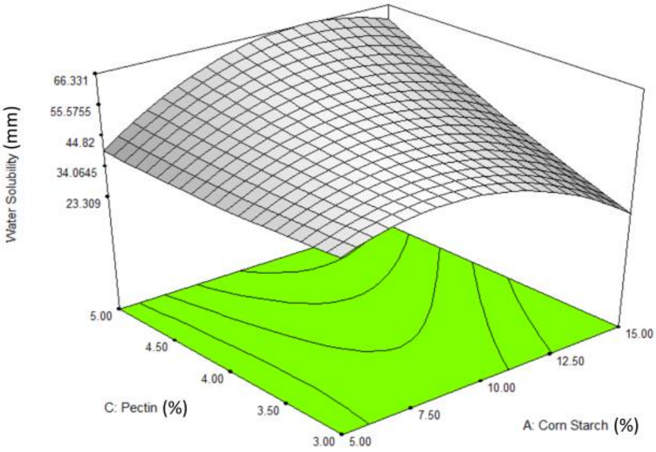


Fig. 5: Water solubility of edible paper at different concertation of corn starch, rice starch and pectin

Mechanical Properties

The mechanical characteristics of various combinations of edible paper are shown in Table -3. These attributes include Tensile Strength, Young's Modulus, and Elongation at Break values. The Tensile Strength readings showed diversity among the tested samples, with a range of 31.678 MPa to 39.413 MPa (Fig. 6 (a)). In addition, the percent of Elongation at Break (Eb) for each combination ranged from 79.143 MPa to 91.765 MPa (Fig. 6 (b)), indicating the degree of deformation before fracture. The observed range of Young's Modulus values, which represent the stiffness and resistance to deformation of the materials, is 931.294 % to 1429.340 % (Fig. 6 (c)). These findings offer a thorough understanding of the mechanical properties of the several edible paper combinations under investigation.

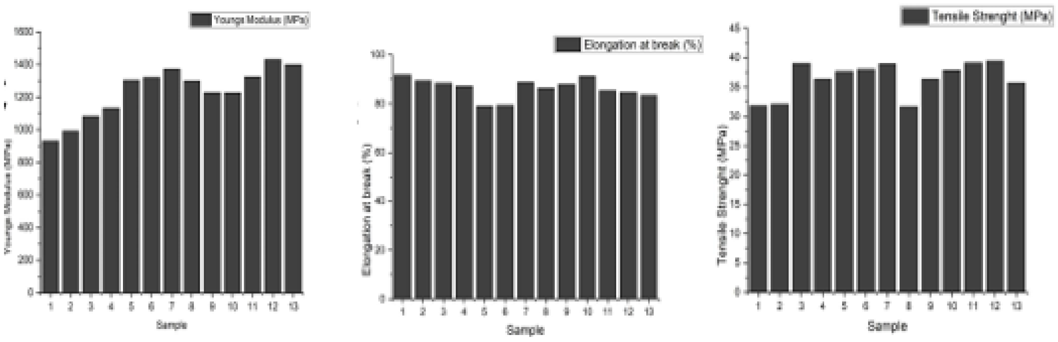
The tensile strength of a film is primarily determined by the intermolecular bonding among its particles. The tensile strength of the packaging material was ranging between 31.678 and 39.413 MPa. Tensile strength of the packaging material was found to be increasing with increase in the concentration of corn starch but effect was not significant ($P<0.05$). This might be due to the formation of thin continuous film on application of pectin. The maximum tensile strength of the edible paper reached 39.413 MPa in sample HT12 {CS (15): RS(10): P(4)}, highlighting the importance of the optimal concentration of corn starch and rice flour in enhancing the material's elasticity and tensile strength. Conversely, sample HT08 {CS(10): RS(10): P(4)} exhibited the lowest tensile strength, possibly attributed to inadequate cross-linking among the dopants. Notably, a study reported a standard tensile strength of approximately 38.22 ± 0.7 MPa for a blend film Zhu, (2021), emphasizing its high performance as a food packaging material, a result consistent with the findings observed in samples HT11 and HT12.

Young's modulus serves as a crucial parameter for assessing a material's stiffness, with a higher Young's modulus value indicating an ideal tensile strength. Variations in pectin and starch concentrations, particularly rice and maize, can influence Young's modulus. Notably, sample HT12 {CS (15): RS(10): P(4)} exhibited the highest Young's modulus, signifying superior flexibility and reduced rigidity. Young's modulus of edible films typically ranges from 350 to 800 MPa, highlighting the material's ability to withstand deformation under stress Jorge et al.,(2023). Samples HT01 and HT02 showed similar results, emphasizing the importance of Young's modulus in determining the mechanical properties of edible food packaging materials.

The elongation at break value of the edible paper, which is based on cornflour and pectin, was assessed alongside the tensile strength and Young's modulus values. This parameter reflects the material's stretchability, determined by the initial length before reaching the breaking point. Edible films can exhibit elongation at break values ranging from 0.976% to 205%, depending on the polymers utilized Maruddin et al.,(2020). Sample HT01 demonstrated results comparable to the established standards, highlighting its mechanical properties in terms of extendibility (Table 3) (Figure 6 and 7).

Table 3. Mechanical properties of starch- pectin based edible paper

Sample code	Tensile Strength (MPa)	Elongation at break (%)	Young’s modulus (MPa)
HT01	31.82	91.77	931.29
HT02	32.10	89.41	992.00
HT03	39.02	88.40	1084.80
HT04	36.34	87.27	1134.10
HT05	37.63	79.14	1304.29
HT06	37.99	79.42	1320.28
HT07	38.93	88.81	1370.84
HT08	31.68	86.48	1299.32
HT09	36.34	87.96	1299.01
HT10	37.85	91.30	1228.22
HT11	39.10	85.38	1324.76
HT12	39.41	84.68	1429.34
HT13	35.72	83.51	1399.26



(a) (b) (c)

Fig. 6: Mechanical properties of edible paper at different concertation of corn starch, rice starch and pectin (a) Tensile strength (MPa) (b) Elongation at break(%) (c) Youngs modulus (MPa)

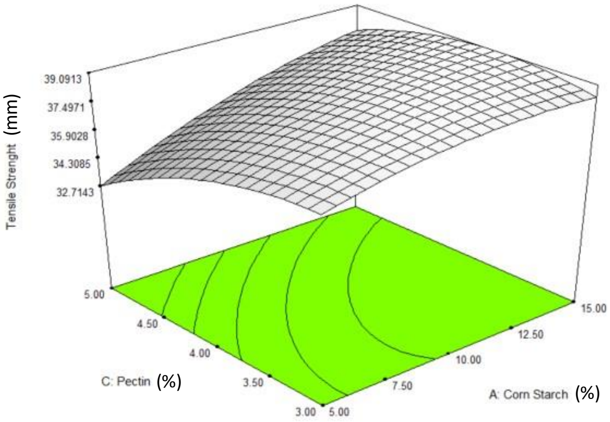


Fig. 7: Tensile strength of edible paper at different concertation of corn starch, rice starch and pectin

X-Ray Diffraction:

The X-ray diffraction (XRD) analysis conducted on pure and processed edible paper samples revealed significant insights into their crystalline and amorphous structures. Initially, the XRD pattern of bare rice starch displayed distinct crystalline peaks at 2θ values of 14.89°, 17.15°, 18.2°, 20.47°, and 23.78°. These peaks are indicative of the inherent crystalline nature of rice starch.

In contrast, the neat composite films, which included various formulations labeled from HT01 to HT13 (Fig. 9), exhibited characteristic peaks that differed from those of pure rice starch. This variation suggests modifications in the crystalline structure due to the addition of other components such as pectin, glycerol, and carboxymethyl cellulose (CMC), which interact with the starch matrix.

The amorphous nature observed in these composite films, particularly in sample HT12 {CS (15): RS(10): P(4)}, which showed peaks at 2θ = 17.36° and 34.29°, can be attributed to the interactions between rice starch and the added dopants. The presence of glycerol and CMC, known for their plasticizing effects, along with pectin, which can form hydrogen bonds with starch, likely disrupts the orderly crystalline arrangement of starch molecules, leading to a more amorphous structure Ibrahim et al.,(2019). This reduction in crystallinity and the resultant amorphous nature enhances the flexibility and moldability of the edible paper, making it more suitable for practical applications where flexibility and durability are required.

The capacity of hydrogen bonding to stabilize the structure by creating a free energy balance also plays a crucial role in the observed phase changes and the decrease in crystallinity Ibrahim et al.,(2019). This interaction between the components within the composite films is essential for achieving the desired mechanical properties in the edible paper, aligning with the requirements for specific food packaging applications (Figure 9 and 10).

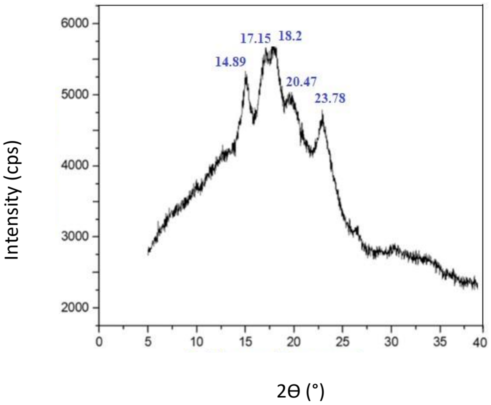
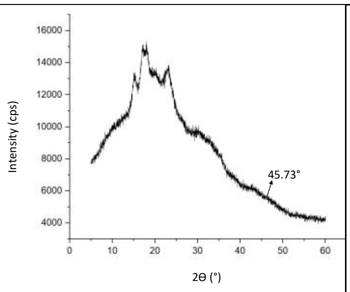
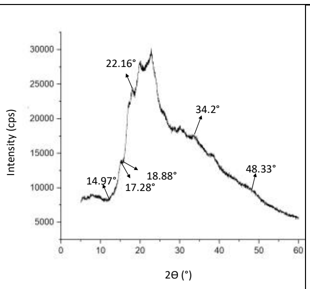


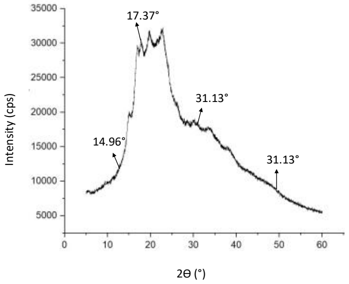
Fig. 8 Rice starch



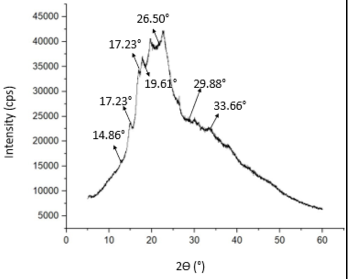
HT01



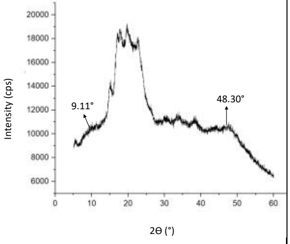
HT02



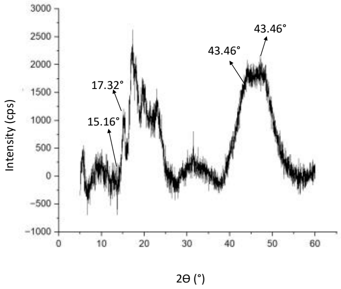
HT03



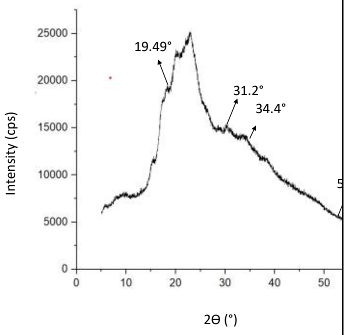
HT04



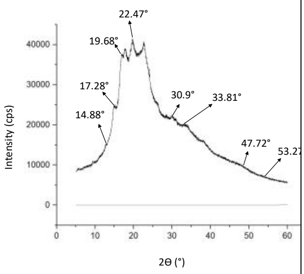
HT05



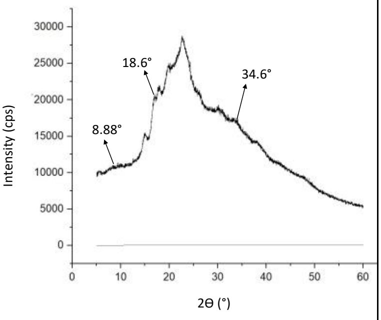
HT06



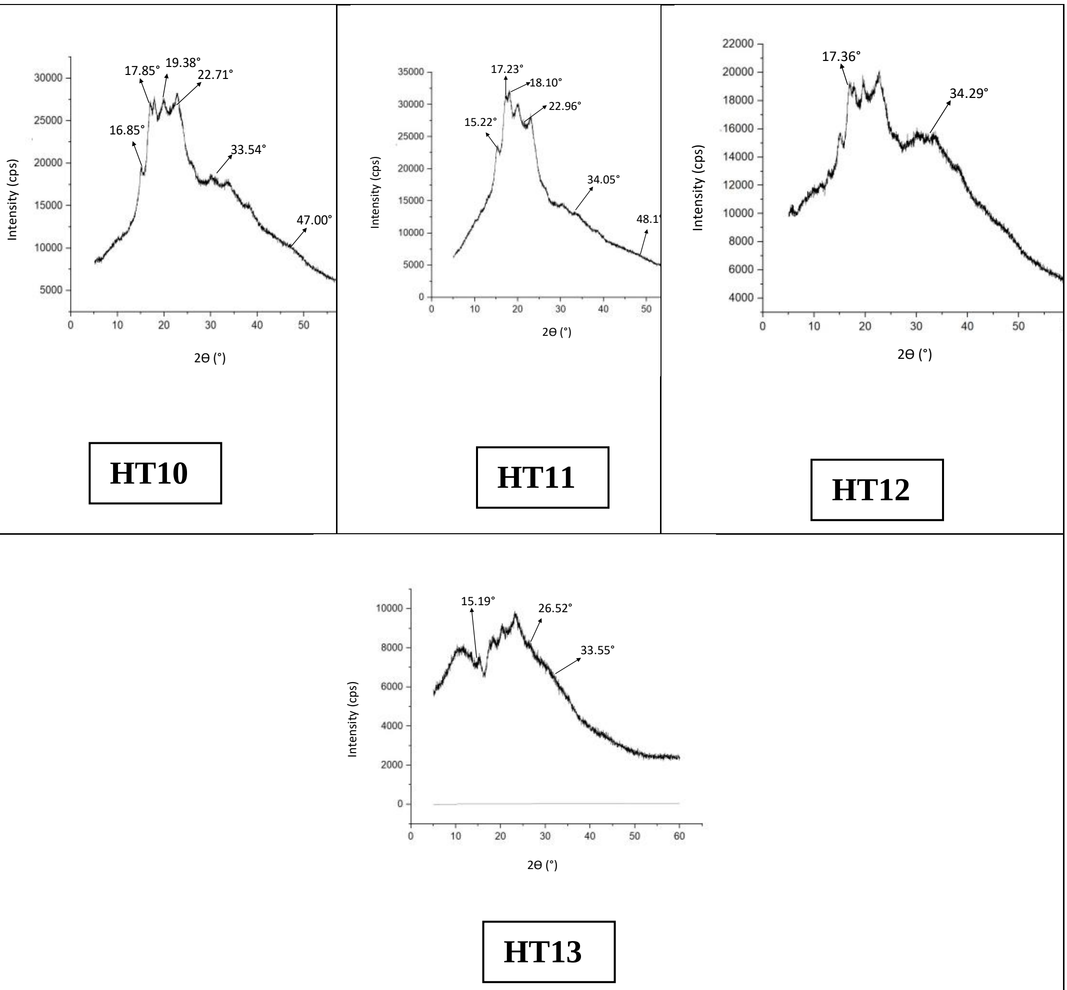
HT07



HT08



HT09



HT10

HT11

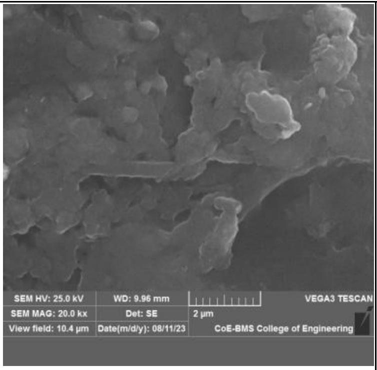
HT12

HT13

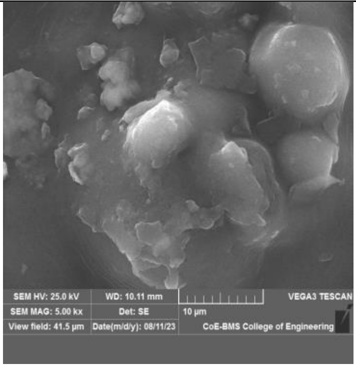
Fig. 9 XRD images of edible paper (HT01- HT13) at different concertation of corn starch, rice starch and pectin 10 SEM images of edible paper (HT01- HT13) at different concertation of corn starch, rice starch and pectin

SEM

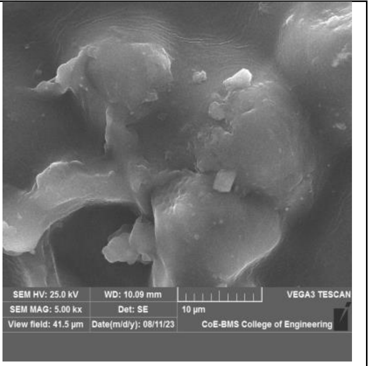
The morphology of the edible paper was analysed through scanning electron microscopy (SEM) from sample HT01-HT13 (Fig. 10), as depicted in the provided figures. The SEM images revealed that certain samples exhibited a non-uniform and heterogeneous surface texture, suggesting inadequate crosslinking among the dopants. However, samples HT12 {CS (15): RS (10): P (4)} and HT10 {CS (15): RS (7.5): P (5)} demonstrated that the edible paper possessed a naturally smooth and uniform texture Kopacic et al.,(2018). The SEM analysis indicated robust cross-linking between the carrier material and the dopants, resulting in the formation of a cohesive and continuous film Liu et al.,(2010) (Figure 10).



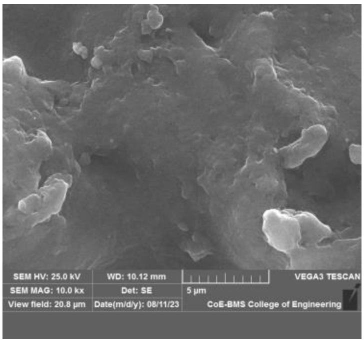
HT01



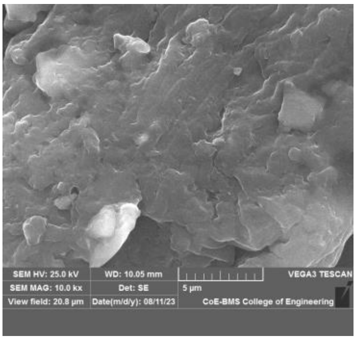
HT02



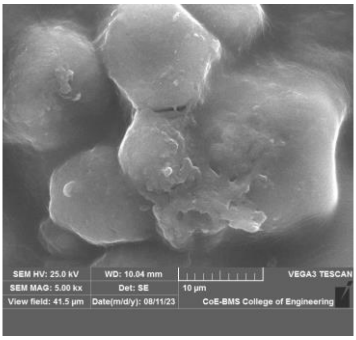
HT03



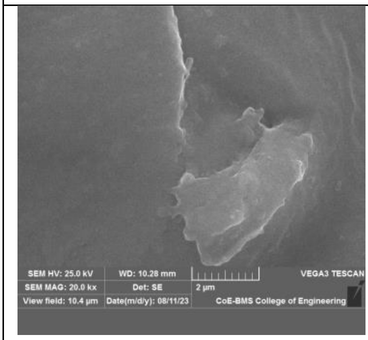
HT04



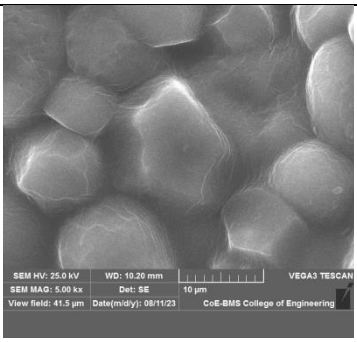
HT05



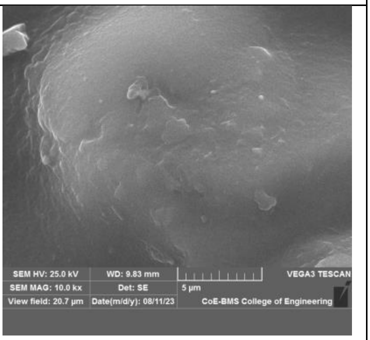
HT06



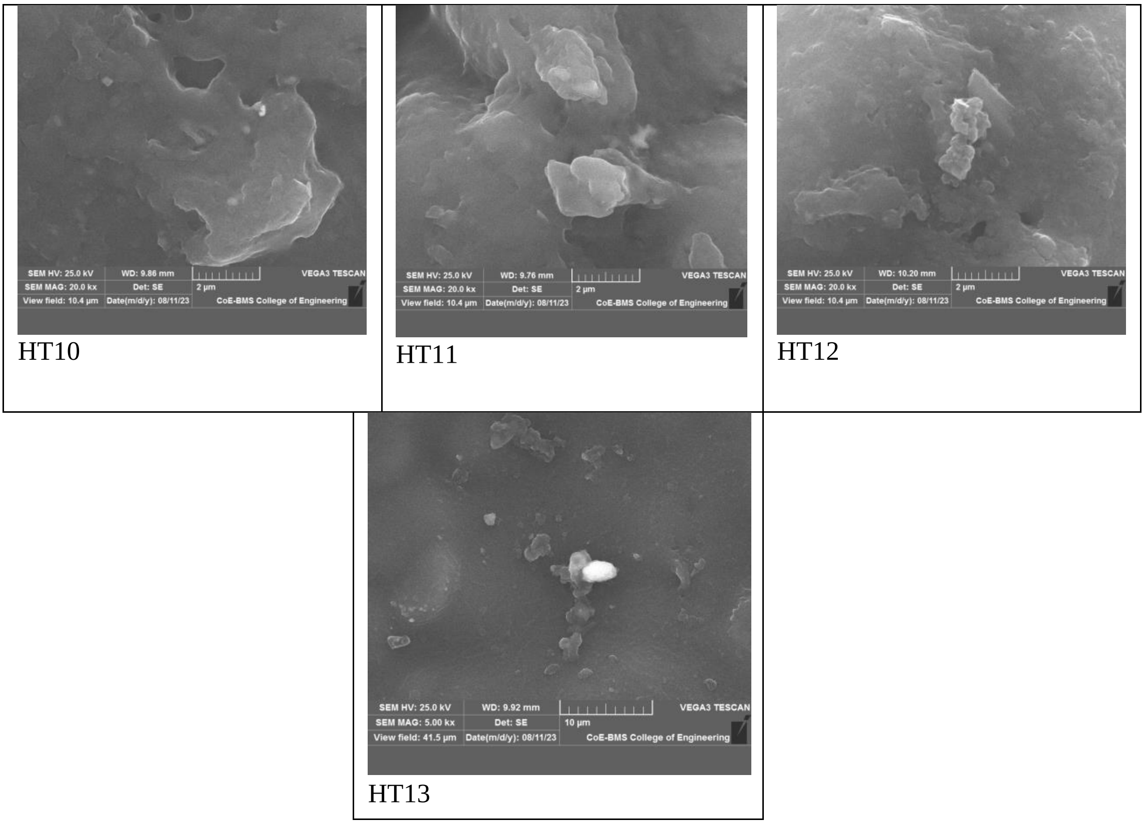
HT07



HT08



HT09



HT10

HT11

HT12

HT13

Fig. 10 SEM images of edible paper (US01- US13) at different concertation of corn starch, rice starch and pectin through the application of ultrasonication

Optimisation of process parameters

The optimization of responses was conducted individually, utilizing outcomes from various experiments and analysis through Design Expert software. Following the interpretation of these results, adjustments were made to the component ratios for further experimentation. Specifically, the range for corn starch was adjusted to 10-15%, rice starch to 7.5-10%, and pectin remained at 3-5%. Additionally, the concentrations of other dopants, including xanthan gum, carboxymethyl cellulose, and glycerol, were maintained at a constant 1%. These revised proportions were employed in subsequent experiments.

4. Conclusion

The focus of this study was on the development of edible paper using corn starch, rice starch, pectin, carboxymethyl cellulose, xanthan gum, and glycerol. The edible paper underwent a series of physical, mechanical, and structural evaluations. The results indicated excellent performance in terms of moisture content and water solubility, which suggested a rapid biodegradability rate. The incorporation of pectin and other dopants with corn starch, serving as the carrier material, facilitated effective cross-linking, contributing to the notable mechanical properties observed. Additionally, the materials were analyzed for their crystalline and amorphous characteristics, with most samples displaying considerable flexibility. All findings were consistent with the ranges established by prior research. Notably, films from HT10 {CS (15): RS(7.5): P(5)} to HT12 {CS (15): RS(10): P(4)} exhibited superior performance, aligning well with established standards. In summary, this research underscores the viability of edible paper as a sustainable option for food packaging, demonstrating its promising mechanical and textural qualities that meet industry benchmarks.

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Ethical approval

Not applicable

Conflict of Interest

Authors declare no conflict of interest.

Data availability

All the data analyzed for this study have been included.

Future Scope

Further, the emulsion can be subjected to ultrasonication to enhance crosslinking and improve the properties of the packaging materials. The effects of ultrasonication can then be evaluated to determine potential modifications and enhancements.

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