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ORIGINAL ARTICLE

Development of novel dabai pulp-based marker ink: optimization of process configurations, physico-chemical properties, and morphological analysis

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Abstract

This work aims to investigate the development and optimization of a novel dabai pulp-based marker ink using response surface methodology (RSM) with a central composite design. The production of dabai pulp-based marker ink was conducted using dabai pulp powder within the range of 50 to 100 g, glycerol from 15 to 62.5 mL, and carboxymethyl cellulose (CMC) between 1 and 12.5 g, aiming to achieve the desired ink properties of viscosity and color lightness. The results from RSM revealed that the optimal conditions for producing dabai pulp-based marker ink were 12.5 g of CMC, 15 mL of glycerol, and 50 g of dabai pulp powder. The viscosity and color lightness of the optimized dabai pulp-based marker ink with glycerol were compared to those of dabai pulp-based ink without glycerol and commercial ink. The results showed that the viscosity and color lightness of the dabai pulp-based marker ink without glycerol and the commercial ink were 542.73 mPa•s and 33.15 L*, and 784.03 mPa•s and 23.92 L*, respectively. Thermogravimetric analysis revealed that the commercial marker ink had the fastest drying rate among the three samples. Fourier transform infrared spectroscopy confirmed the presence of functional groups associated with the dabai pulp-based marker ink. Morphological analysis indicated that dabai pulp powder with glycerol exhibited aggregation, resulting in a much smoother and flatter surface. Overall, dabai pulp-based marker ink shows promise as a viable and innovative material for non-food applications.

Keywords Dabai · Marker ink · Optimization · Response surface methodology · Characterization

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1 Introduction

Dabai (*Canarium odontophyllum*) has become a highlight among the many unique fruits grown on Borneo Island, particularly in Sabah and Sarawak, Malaysia [1]. During its fruit season, the dabai fruit has gained popularity among Sarawak's Iban community, which has led to a dedicated festival called "Pesta Dabai" that is held annually by the Kapit District Council. In addition to being native to Sarawak, dabai fruits can also be discovered growing along riverbanks in Sibu, Kapit, and Sarikei divisions of the state [2]. Over the past few years, many studies have been carried out on dabai, examining the physical and chemical properties of the pulp, kernel, leaf, and bark of the plant [3–5]. Dabai contains nutrients such as 4-hydroxybenzoic acid, syringic acid [6], anthocyanin, phosphorus, iron, potassium, magnesium, and calcium [7].

Dabai has been recognized for its unique qualities and cultural significance. Dabai has been utilized in various food-based and water-processing-related products using its pulp, peel, and seed, including essential oil products [8], and serve as a precursor to the production of biocarbon for wastewater treatment [9], cookies [10], and cocoa bar products [11]. The focus has now shifted toward exploring its potential for non-food applications. In this context, the production of marker ink has garnered significant interest from researchers. It has been demonstrated that marker ink can be produced from a variety of agricultural products, such as berry seed [12], mangosteen leaves [13], and neem trunk [14]. To the best of our knowledge, no research has been conducted on using dabai pulp in marker ink.

Currently, ink production relies heavily on non-renewable synthetic resources and chemicals, including solvents such as 2-pyrrolidone, propanediol, glycols, and phthalates, all derived from petroleum and chemicals [15, 16]. These substances can have harmful environmental and health impacts on both users and ecosystems. A person exposed to organic solvents in ink-related industries can exhibit symptoms such as fatigue, irritability, and memory impairment, in addition to neurobehavioral deficits such as impaired motor performance and information processing [17]. Solvent-based inks are known for their high durability and fast drying times, but they release volatile organic compounds. In recent years, the trend has shifted toward reducing the volatility and enhancing the health and safety properties of these inks [18]. These issues provide the following insights for future research in producing natural source or biobased ink [19]. Even though many researchers have worked on the utilization of agricultural resources to manufacture marker ink and its characterization, the determination of the process configuration for dabai pulp-based marker ink production in terms of quality has yet to be achieved.

Inks serve a variety of purposes, including printing, coloring, and writing, with different formulations tailored to meet specific needs and conditions. The primary raw materials used in ink production include pigments, binders, solvents, and additives [20]. Several reports have been published to date regarding the use of carboxymethyl cellulose (CMC) and glycerol as binding agents or additives in the production of marker ink. Biswas et al. demonstrated the digital printing of tyrosinase enzyme on plasma-treated polyamide-6,6 fabric using various sustainable technologies. Among the ink formulations tested, one containing CMC was found to be the most effective viscosity modifier for the enzyme [21]. Indeed, Nocheseda et al. [22] introduced a safer, more sustainable, and cost-effective metallic ink composed of inexpensive, irregularly shaped metallic powders, a biodegradable cellulose-derived polymer (sodium-CMC), guar, bentonite, and deionized water as the solvent.

Similarly, Ding et al. [23] demonstrated that CMC-based edible inks exhibited excellent printing quality, as evidenced by their favorable pH, scratch resistance, optical density, contact angle, and chromatic values on coated paper after both flexographic and screen printing. In addition to their impressive printability, these inks also displayed significant antibacterial activity against *Escherichia coli* and *Staphylococcus aureus*. In a study aimed at enhancing the physical and rheological properties of ink, as well as the print quality on cotton fabric, inks containing glycerol, diethylene glycol, and triethylene glycol have humectants with high boiling points but demonstrated slower evaporation rates [24]. While these inks improved evaporation control, they led to a decrease in both the storage stability of the pH value of the ink and the dye fixation quality of the printed fabric. According to Basri et al. [13], the optimal formulation for the novel mangosteen-leaf-based marker ink is achieved when glycerol, benzalkonium chloride, ferrous sulfate, and CMC are used in the following weight percentages: 5%, 5%, 1%, and 3%, respectively. A small amount of glycerol was found to increase the color lightness value, while a reduction in CMC concentration led to a decrease in the viscosity of the marker ink.

Overall, this approach not only addresses environmental concerns but also promotes a user-friendly formulation of marker ink by replacing traditionally used commercial marker ink with plant-based alternatives. This not only reduces the environmental impact of the ink but also ensures an eco-friendly formulation characterized by non-toxicity and improved safety for users [25, 26]. Understanding the formulation process of ink and their effects on marker ink quality has been a key area of research. Various methods have been employed to test ink qualities, focusing on essential properties such as viscosity color and drying time. For example, a study on berry-based marker inks shows how these properties affect the ink's functionality, with viscosity and drying time being keys for proper application and usability [12]. Studies show that viscosity and drying time are keys to whiteboard ink performance [14]. A balance is needed where too high or low viscosity can clog markers or cause uneven application, while drying time must be quick enough to prevent smudging but slow enough for proper adhesion. Meanwhile, herbal inks from plant sources like bio-indigo and quebracho red were tested for viscosity and density [15]. These properties, along with color and texture, are keys to evaluating the ink's performance and printability. However, not all had comparable accuracies due to the differences in physico-chemical properties of the materials used. Therefore, this study aims to explore the potential of dabai as an alternative, sustainable resource for developing eco-friendly and cost-effective ink. The objective of this study is to develop a dabai pulp-based marker ink by optimizing its formulation using response surface methodology (RSM) and evaluating its physico-chemical and morphological properties in comparison with commercial ink.

2 Materials and method

2.1 Materials

A total of 20 kg of matured dabai fruits (*Canarium odontophyllum*) were sourced from a local supplier in Song, Sarawak, Malaysia. The chemical reagents used in this study included CMC, ferrous sulfate, benzalkonium chloride, and glycerol, all of which were purchased from DChemie, Malaysia. In addition, a commercial marker ink (Artline, Schachihata, Japan) was procured from a local bookstore in Selangor to serve as a reference sample for comparative analysis.

2.2 Flowchart of methodology

Figure 1 presents the overall flowchart of the methodology for this study. The methodology was structured into several key steps to optimize the production and characterization of novel dabai pulp-based marker ink.

2.3 Sample preparation

First, the dabai fruits were wrapped with newspapers, packed in an ice box, and delivered to Universiti Putra Malaysia within 24 h after wrapping. Upon arrival, they were stored in a cold room at 4 °C (SIA291583, Thematic A35900, Malaysia) to ensure freshness and prevent spoilage prior to processing [8]. In the initial stage of sample preparation, the dabai pulp was meticulously peeled away from its skin and allowed to dry. As soon as the pulp was peeled, it was placed on the tray and then dried in a box dryer (WS 30, WA, USA) at 60 °C for 3 days [8]. Following that, the dried dabai pulp was crushed by a mortar and pestle to achieve the desired chunky texture. Then, the samples were ready to be used for the experiment.

2.4 Preparation of dabai pulp-based marker ink

The dabai pulp-based marker ink was prepared according to the protocol described with a few modifications [13]. The desired amount of dabai powders was weighed using a digital weighing scale (Mettler Toledo, Columbus, OH, USA). Following that, 375 mL of distilled water, which was fixed for all experiments, and the required quantity of glycerol, which varied between 1.19 and 78.69 mL depending on the experimental design in Table 1, were mixed in a beaker and vigorously swirled for 30 s. The mixture was boiled together on a hot plate (Thermo Fisher Scientific, Waltham, MA, USA) at a temperature heating degree of 8 (circa 300 °C) for 30 min to improve the dye extraction rate from dabai pulp. In order to reduce vapor evaporation, aluminum foil was placed over the beaker. Upon boiling, the temperature was reduced to a heating degree of 2 (circa 100 °C). After the mixture had been boiled for some time, the hot plate was turned off, and the mixture was collected in a container using a sieve to remove the dabai pulp powder. Only 50 mL of the dabai ink was collected for further testing. Prior to testing, the collected dabai pulp-based marker ink was stored in an airtight glass container for 24 h in a cool and dry place to allow for settling and ensure its stability.

2.5 Optimization of dabai pulp-based marker ink production by response surface methodology (RSM)

The Design Expert 11.1.2.0 software was used to optimize the effect of dabai pulp powder, glycerol, and CMC as independent variables on the marker ink production from dabai pulp. The interaction results and optimum parameters were obtained. Table 1 presents that the RSM model experimental data values and their encoded levels. The process parameters' actual levels and ranges were expressed as – 1 (low), 0 (center), + 1 (high), + α (maximum), and – α (minimum). The dabai pulp-based marker ink production was produced at dabai pulp ranging from 50 to 100 g, glycerol of 15 to 62.5 mL, and CMC of 1 to 12.5 g. It is important to note that the CMC factor initially had a negative axial value of – 2.92 g. However, since CMC cannot practically have a negative value (as it is a mass-based component), the axial value for CMC was adjusted to + 2.92 g to ensure the feasibility and symmetry of the design. Table 2 shows the experimental design for the 20 runs that were performed in order to produce dabai pulp-based marker ink using a three-level central

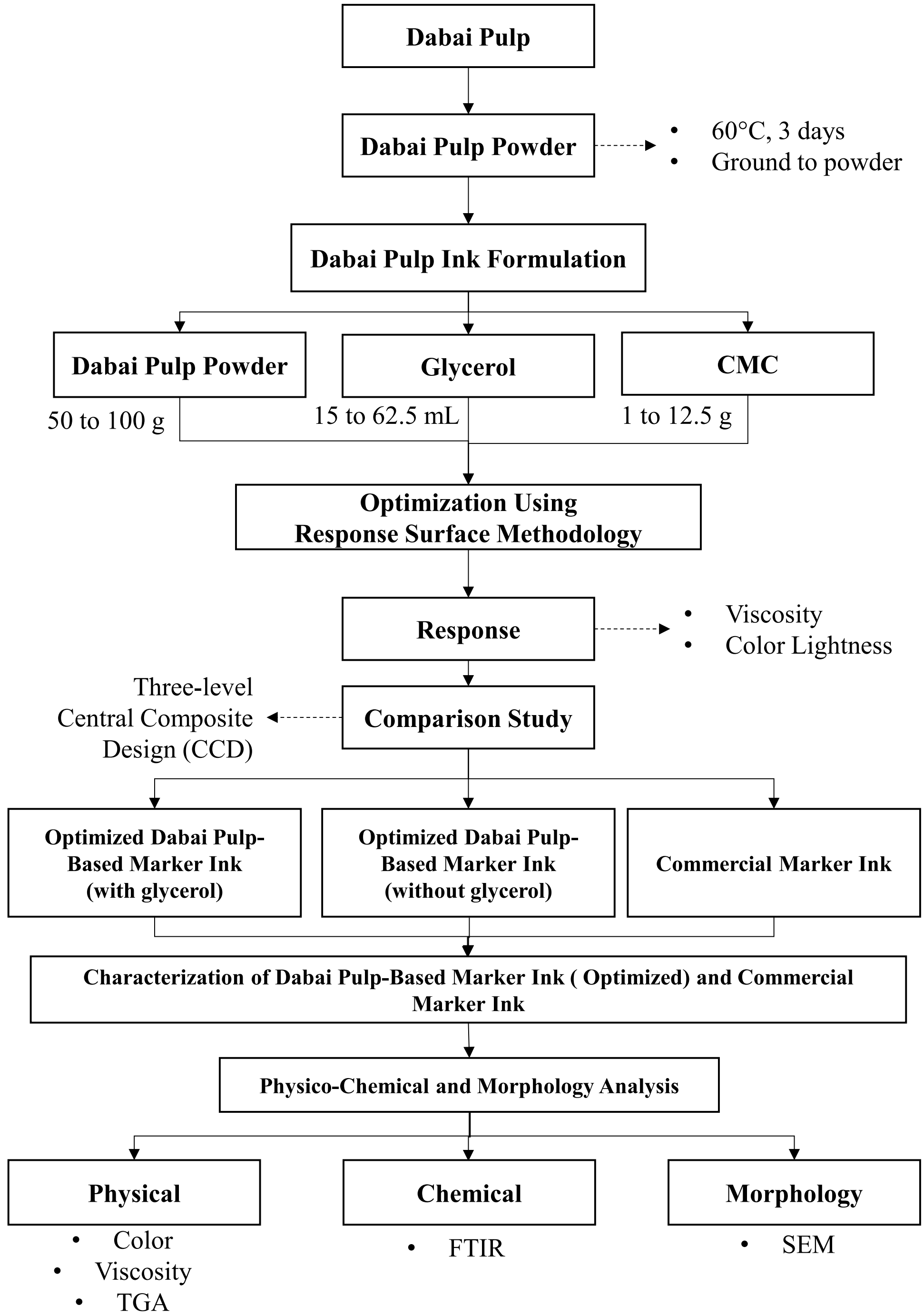

Fig. 1 The flowchart of the methodology

Table 1 The RSM model experimental data values and their encoded levels

Notation	Parameters	Coded levels				
		−α	−1	0	1	+α
A	Dabai pulp powder (g)	32.96	50	75	100	117.04
B	Glycerol (mL)	1.19	15	38.75	62.5	78.69
C	CMC (g)	2.92	1	6.75	12.5	16.42

Table 2 The independent variables and responses of viscosity and color

Run	Independent variables			Response variables	
	Dabai pulp powder (g)	Glycerol (mL)	CMC (g)	Viscosity (mPa•s)	Color (L*)
1	75.00	38.75	6.75	2102.93	28.87
2	50.00	15.00	12.50	659.23	36.80
3	50.00	62.50	12.50	795.93	35.20
4	75.00	79.00	6.75	716.47	29.47
5	117.05	38.75	6.75	544.00	32.80
6	50.00	62.50	1.00	80.77	28.20
7	100.00	62.50	1.00	64.47	29.13
8	75.00	38.75	2.92	1574.27	28.13
9	75.00	38.75	6.75	2102.93	28.87
10	75.00	38.75	6.75	2102.93	28.87
11	75.00	38.75	6.75	2102.93	28.87
12	50.00	15.00	1.00	14.90	29.87
13	100.00	62.50	12.50	549.95	32.83
14	75.00	1.20	6.75	449.70	34.00
15	75.00	38.75	6.75	2102.93	28.87
16	100.00	15.00	12.50	820.17	36.80
17	75.00	38.75	6.75	2102.93	28.87
18	32.90	38.75	6.75	53.43	30.17
19	75.00	38.75	16.42	577.00	33.53
20	100.00	15.00	1.00	555.00	31.20

composite design (CCD). Each run was performed in triplicate. The descriptive statistics and statistical tests of the experimental work were analyzed using analysis of variance (ANOVA).

2.6 Physico-chemical properties of dabai pulp based-marker ink and morphology characterization of dabai pulp powder

The physicochemical properties of the optimized dabai pulp-based marker ink (with glycerol) were compared to those of the dabai pulp-based marker ink (without glycerol) and commercial marker ink. The microstructure of dabai pulp powder was characterized for morphology properties.

2.6.1 Viscosity

The marker ink samples were evaluated to ensure optimal performance, with testing conducted 24 h after preparation. Viscosity measurements were performed using a Model RDVD-II Brookfield rotational viscometer (Brookfield Engineering Inc., Middleboro, MA, USA) fitted with an L2 fixed spindle, following the modified method originally outlined by Bozdogan [27].

2.6.2 Color

The CIE color analysis of marker ink samples was observed using HunterLAB Ultrascan, Pro d/8 Spectrophotometer (Hunter Associated Laboratory, Inc, Reston, VA, USA) as described by Kanokruangrong et al. [28]. A 200 mL of marker ink sample was placed in front of the porthole, and the three-dimensional coordinates L* (lightness), a (red/green value), and b (blue/yellow value) were captured.

2.6.3 Thermogravimetric analysis

Thermogravimetric analysis was performed on marker ink samples using a Mettler Toledo AG-TGA/STDA (Model 851e, Mettler Toledo AG, Greifensee, Zurich, Switzerland) based on the method reported by Basri et al. [13]. For each test, 20 mg of ink was prepared. Nitrogen was used as the medium, and the samples were heated from 20 to 200 °C at a rate of 25 °C/min. Three ink samples, representing optimized dabai pulp-based marker ink (with and without glycerol) and commercial marker ink, were tested to observe variations in their drying rates. As the drying process continued, the weight of the sample decreased in proportion to solvent evaporation. The drying rate of a sample is the weight of solvent lost per unit of time divided by the evaporation area [29]. Solvent loss over time was monitored by tracking changes in mass, as recorded by the balance. The plot of weight percentage against time was generated.

2.6.4 Fourier transform infrared spectroscopy (FTIR)

The marker inks were analyzed using FTIR spectroscopy (PerkinElmer Spectrum 100, USA) at a resolution of 4 cm^{-1}. The spectra were recorded in the range of 4000 to 500 cm^{-1} [30]. Three samples were examined: commercial ink, optimized dabai pulp ink with glycerol, and optimized dabai pulp ink without glycerol.

2.6.5 Scanning electron microscope (SEM)

Raw dabai pulp powder, optimized dabai pulp powder with glycerol, and optimized dabai pulp powder without glycerol were evaluated and compared at × 250 magnification using a scanning electron microscope (SEM) (S3400N, Hitachi, Japan). The samples were gold-coated for 120 s (Quorumtech EMscope SC500 Sputter Coater, UK) before the examination. The photos were taken under vacuum conditions using an accelerating voltage of 15 kV.

3 Results and discussion

3.1 Optimization of dabai pulp-based marker ink by response surface methodology (RSM)

Table 2 presents the experimental values of viscosity and color (L*) for three key parameters: dabai pulp powder (g), glycerol (mL), and CMC (g). This table illustrates the optimization process conducted using RSM to determine the optimal levels of these parameters for formulating dabai pulp-based marker ink. A highest-order polynomial model was employed to interpret and represent the significance of the response components, evaluated through the sequential model sum of squares analysis. The validity of the quadratic model was confirmed by the R^2 value, which helped identify the optimal parameters for modifying the viscosity and color of the marker ink.

Based on Table 2, the lowest viscosity recorded was 14.90 mPa•s in the 12th trial, which used 50 g of dabai pulp powder, 15 mL of glycerol, and 1 g of CMC. Conversely, the highest viscosity observed was 2102.93 mPa•s, achieved at the center point design with 75 g of dabai pulp powder, 38.75 mL of glycerol, and 6.75 g of CMC. For color lightness (L*), the minimum value observed was 28.13 in the eighth trial, which used 75 g of dabai pulp powder, 38.75 mL of glycerol, and 2.92 g of CMC. In contrast, the maximum value recorded was 36.80, observed in both the second trial, with 50 g of dabai pulp powder, 15 mL of glycerol, and 12.5 g of CMC, and the 16th trial, which used 100 g of dabai pulp powder, 15 mL of glycerol, and 12.5 g of CMC.

From these results, it can be concluded that the combination of higher dabai pulp powder, moderate glycerol, and higher CMC positively influenced the marker ink's viscosity and color lightness. Higher dabai pulp powder increased the pigment concentration, resulting in higher viscosity and better color quality. However, the development of pigment-based inks continues to face several challenges, including particle size requirements, dispersion stability, and the need for an effective binder [31]. CMC is an ideal binder for formulating conductive printable inks due to its unique properties, including thickening, stabilizing, and binding properties [24]. In pigment and color extraction, glycerol serves as a solvent, stabilizer, and humectant, enhancing the solubility, yield, and stability of the pigments. Its role is especially crucial when extracting hydrophilic pigments like anthocyanins, ensuring a higher yield and maintaining the quality and intensity of the extracted color [32]. In addition, temperature plays a critical role in the extraction process [33]. While higher temperatures can increase the solubility of pigments, they may also lead to a decrease in yield if the temperature is too high. Glycerol helps mitigate the negative effects of temperature by stabilizing the pigments and preventing degradation, thereby optimizing the overall extraction process [13]. Therefore, one aspect to consider for further study is the careful selection and optimization of the extraction temperature to ensure the stability of the ink. These characteristics of ink enable effective control of ink viscosity, ensuring optimal performance across various applications. These results should be interpreted with caution due to the potential influence of various factors, including uncontrolled variables and biases that may affect the outcome. A more detailed discussion of the interaction between variables and their effects on the response variables is provided in the following section.

3.2 Interaction between independent variables on viscosity and color

Three-dimensional (3-D) surface plots and two-dimensional (2-D) contour plots were utilized to determine the optimal formulation variables for marker ink from dabai pulp. As shown in Fig. 2(a), increasing the amount of dabai pulp powder from 50 to 100 g and glycerol from 15 to approximately 45 mL leads to a notable increase in viscosity, reaching a peak of around 2000 mPa·s. Similarly, as shown in Fig. 2(b), increasing the dabai pulp powder from 50 to 100 g and CMC from 1 to approximately 10.2 g also leads to a higher viscosity, with an optimal peak reaching around 2000 mPa·s. In Fig. 2(c), as glycerol concentration increases from 15 to 45 mL, along with CMC from 1 to approximately 10.2 g, viscosity initially rises to around 2000 mPa·s. Still,

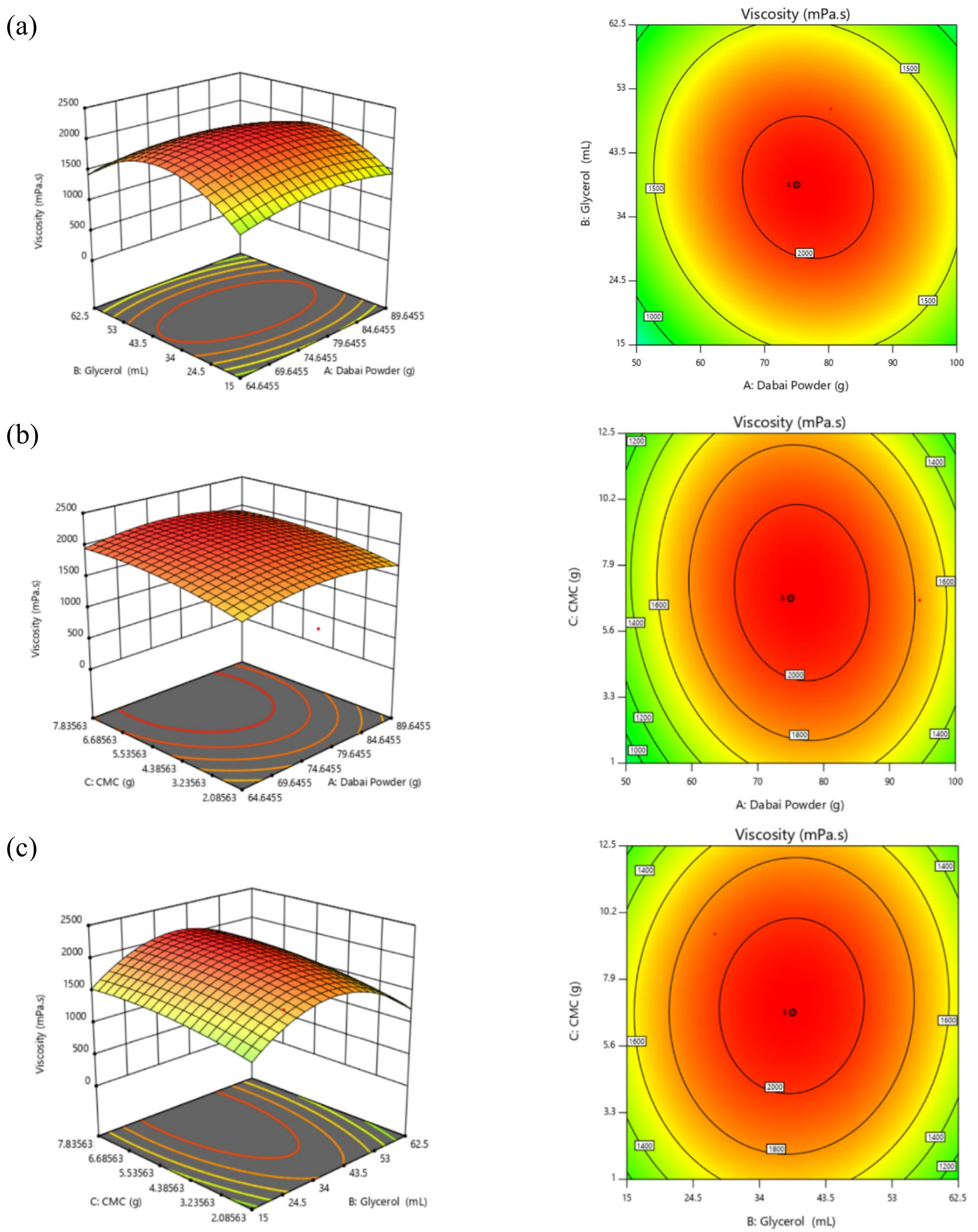

Fig. 2 The surface and contour plots of dabai marker ink; interaction between (**a**) dabai powder and glycerol, **b** dabai powder and CMC, and (**c**) glycerol and CMC for viscosity

it then stabilizes, without a significant decline at higher concentrations.

Across all figures, a consistent pattern emerges in which viscosity increases with the quantity of components up to an optimal point, after which it begins to slightly decline. Increasing dabai powder with either glycerol or CMC increases viscosity, making the ink thicker. While increasing CMC also leads to an increase in viscosity, indicating that CMC is a strong thickening agent. This study highlights the importance of a balanced powder, CMC, and glycerol formulation in achieving and maintaining the desired viscosity levels. According to Fazriyah et al. [34], achieving the right balance of ingredients is critical for producing high-quality marker ink that prevents clogs or excessive spreading. The optimized viscosity obtained using RSM was 357.72 mPa•s. To validate the accuracy of this model, it is essential to conduct validation under the optimal conditions predicted by RSM. Additionally, the quadratic model was evaluated to determine the best correlation with the quantities of dabai pulp powder glycerol and CMC. The viscosity of the marker ink plays a crucial role in its flow behavior and overall performance. The interaction between dabai pulp powder, glycerol, and CMC significantly influences the ink's viscosity, thereby affecting its functionality. Lower glycerol and higher dabai levels decrease viscosity, as shown in the plot (Fig. 2(a)). Similar findings have been reported in other studies, where adjusting the levels of components such as CMC significantly altered the viscosity of marker inks [13].

Lazuardi and Haryanto [35] conducted a related study examining the effects of natural pigments, binder, and their interactions on ink properties using alcohol, coloring from dragon fruit peels, water, and resin (gum Arabic). As the concentration of gum Arabic decreases, the ink density becomes lower. Meanwhile, the drying speed is directly proportional to the ink's viscosity. Higher viscosity results in slower drying time. This finding aligns with the data presented by Rahayu and Fatimah [36], who investigated the effect of varying coconut shell carbon concentrations on the properties of eco-friendly whiteboard marker inks. Their study showed that increasing the carbon concentration resulted in higher viscosity and density of the ink. Similarly, the current study on dabai pulp explores how the interaction between dabai pulp powder, glycerol, and CMC affects viscosity. Both studies highlight the importance of carefully balancing ingredients to optimize ink properties. Optimizing the color lightness of marker ink involves carefully adjusting the levels of glycerol, dabai powder, and CMC. Figure 3 presents the 3D graph and contour plot illustrating the interaction of process parameters on color.

In Fig. 3(a), it can be observed that as the glycerol concentration decreases from 62.5 to 15 mL and the dabai powder increases from 50 to 100 g, the ink becomes progressively lighter in color. This suggests that a lower glycerol content combined with a higher amount of dabai powder leads to a lighter ink and an increase in L* value. In Fig. 3(b), increasing the CMC concentration from 1 to 12.5 g while varying the dabai powder content between 50 and 100 g also results in greater color lightness. This indicates that reducing the amount of dabai powder and increasing CMC contribute to a lighter shade. Similarly, in Fig. 3(c), when glycerol decreases from 62.5 to 15 mL and CMC increases from 1 to 12.5 g, the color lightness becomes more pronounced. As glycerol decreases and dabai powder increases, the ink becomes lighter (higher L* value). Similarly, increasing CMC also lightens the ink, with the combination of lower glycerol and higher CMC further enhancing color lightness. These findings suggest that for optimal color lightness, it is crucial to reduce glycerol and increase CMC. This balance ensures the ink achieves the desired lighter color, maintaining both consistency and quality in the final product.

The color of marker ink is essential for its visual quality. Studies have shown that the right mix of various ingredients can produce the best color quality to achieve the optimum printing inks to be printed on different substrates [37–39]. Another study found that changing the levels of additives in ink formulations can improve color strength, color fastness, and pilling resistance [40]. The findings of this study align with the results presented in the manuscript by Basri et al. [13]. The study demonstrates that the ink formulation, particularly the selection and concentration of viscosity modifiers like CMC, plays a critical role in determining the ink's color characteristics. This is because CMC helps disperse pigments evenly and maintain their stability, preventing clumping and sedimentation, which could otherwise dull the color intensity. In addition, this adhesive binder improves the ink's adhesion to surfaces, enhancing both color brightness and consistency, while also providing fire retardancy properties [41].

3.3 Statistical analysis

Based on the following second-order polynomial equations, multiple regression analysis revealed a relationship between the response variable and the test variables. Equation 1 expresses viscosity, while Eq. 2 represents color, both as functions of the coded independent variables:

$$\begin{aligned}\text{Viscosity of dabai pulp} - \text{based marker ink (mPa.s)} &= 2106.87 + 92.54\,(A) \\ &- 8.02\,(B) + 31.70\,(C) \\ &- 120.41\,(A*B) \\ &- 76.11\,(A*C) + 36.39\,(B*C) \\ &- 663.62\,(A^2) - 563.08\,(B^2) \\ &- 388.94\,(C^2)\end{aligned} \tag{1}$$

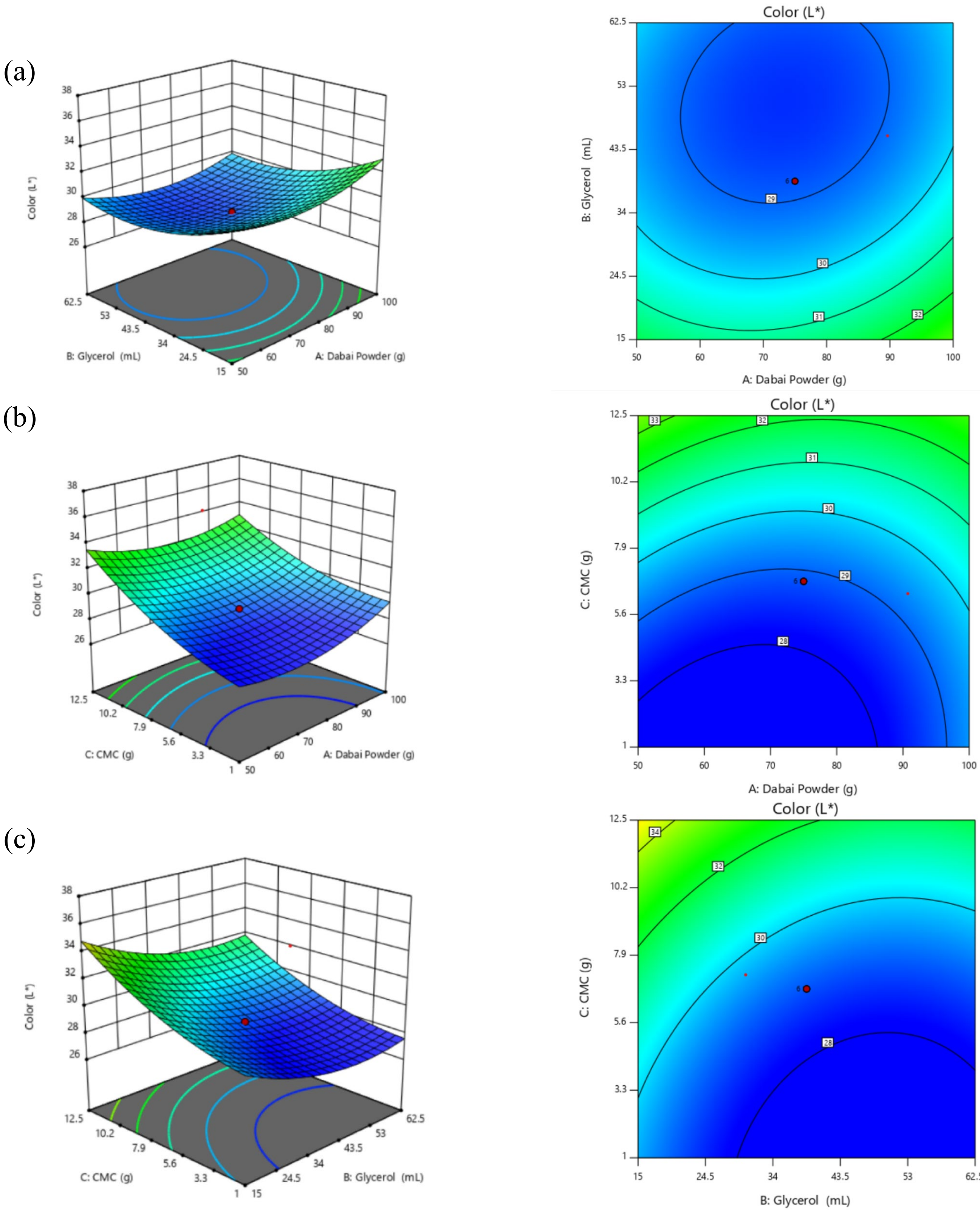

Fig. 3 The surface and contour plots of dabai marker ink; interaction between (**a**) dabai powder and glycerol, **b** dabai powder and CMC, and (**c**) glycerol and CMC in color

Table 3 ANOVA table for quadratic models for viscosity and color

Viscosity							Color						
Source	Sum of squares	df	Mean square	*F*-value	*P*-value		Source	Sum of squares	df	Mean square	*F*-value	*P*-value	
Model	1.138×10^7	9	1.265×10^6	10.62	0.0005	Significant	Model	146.57	9	16.29	14.16	0.0001	Significant
A—Dabai pulp powder	1.169×10^5	1	1.169×10^5	0.9815	0.3452		A—Dabai pulp Powder	1.37	1	1.37	1.19	0.3003	
B—Glycerol	878.56	1	878.56	0.0074	0.9333		B—Glycerol	20.97	1	20.97	18.24	0.0016	
C—CMC	13,725.65	1	132,725.65	0.1152	0.7413		C—CMC	76.46	1	76.46	66.50	<0.0001	
AB	1.160×10^5	1	1.160×10^5	0.9735	0.3471		AB	0.9568	1	0.9568	0.8321	0.3831	
AC	46,337.29	1	46,337.29	0.3889	0.5468		AC	2.68	1	2.68	2.33	0.1576	
BC	10,596.04	1	10,596.04	0.0889	0.7716		BC	0.4201	1	0.4201	0.3654	0.5590	
A^2	6.347×10^6	1	6.347×10^6	53.27	<0.0001		A^2	18.63	1	18.63	16.20	0.0024	
B^2	4.569×10^6	1	4.569×10^6	38.35	0.0001		B^2	21.64	1	21.64	18.82	0.0015	
C^2	2.180×10^6	1	2.180×10^6	18.30	0.0016		C^2	11.86	1	11.86	10.31	0.0093	
R^2	0.9053						R^2	0.9273					

$$\text{Color lightness of dabai pulp} - \text{based marker ink } (L*) = 28.83 + 0.3170\,(A) - 1.24\,(B) + 2.37\,(C) - 0.3458\,(A*B) - 0.5792\,(A*C) - 0.2292\,(B*C) + 1.14\,(A^2) + 1.23\,(B^2) + 0.9071\,(C^2) \quad (2)$$

where dabai pulp powder (A), glycerol (B), CMC (C), interaction factors (AB, BC, AC), and second-order effect (A^2, B^2, and C^2) are considered, the ANOVA is used to determine if the process parameters are statistically significant, as shown in Table 3. Model analysis and coefficient of determination (R^2) analysis are also used to evaluate the suitability of the models. All results were calculated from the average of three trials.

For viscosity, the model *F*-value is 10.62, and for color, the model *F*-value is 14.16, indicating that both models are statistically significant. The *p*-value for viscosity is 0.0005, and for color, it is 0.0001. Since both *p*-values are less than 0.05, the model terms are considered significant. Parameter A (dabai pulp powder) has the greatest impact on viscosity, with an *F*-value of 0.9815. For color, parameter C (CMC) has the most significant effect, with an *F*-value of 66.50.

For viscosity, dabai pulp powder (model A) had the highest *F*-value of 0.9815, showing the greatest impact, followed by CMC (model C), with an *F*-value of 0.1152. Conversely, glycerol (model B) also had a low *F*-value of 0.0074. This ranking suggests that dabai pulp powder is the most influential parameter, with CMC also contributing significantly, whereas glycerol has the least impact. For color, CMC (model C) had the highest *F*-value of 66.50, followed by glycerol (model B) with an *F*-value of 18.24, and dabai pulp powder (model A) with a lower *F*-value of 1.19. The *p*-value analysis showed model A's *p*-value at 0.3003, exceeding the 0.1000 threshold, suggesting it was not significant for this experiment. Both the viscosity and color of dabai marker ink have high coefficients of determination (R^2), which indicates that the models used to predict these properties are highly effective. For viscosity, the R^2 value is 0.9053, showing the model explains 90.53% of the variability in the ink's viscosity. Similarly, for color, the R^2 value is 0.9273, meaning the model explains 92.73% of the variability in the ink's color.

Figure 4(a) and (c) displays the predicted versus actual plots, while Fig. 4(b) and (d) presents the normal probability plots of the residuals for viscosity and color, respectively. Figure 4(a) and (c) shows predicted vs. actual plots that are approximately linear, suggesting that the residuals follow a normal distribution. For viscosity (Fig. 4(a)), though there are outliers, the high R^2 values of 0.9053 as previously

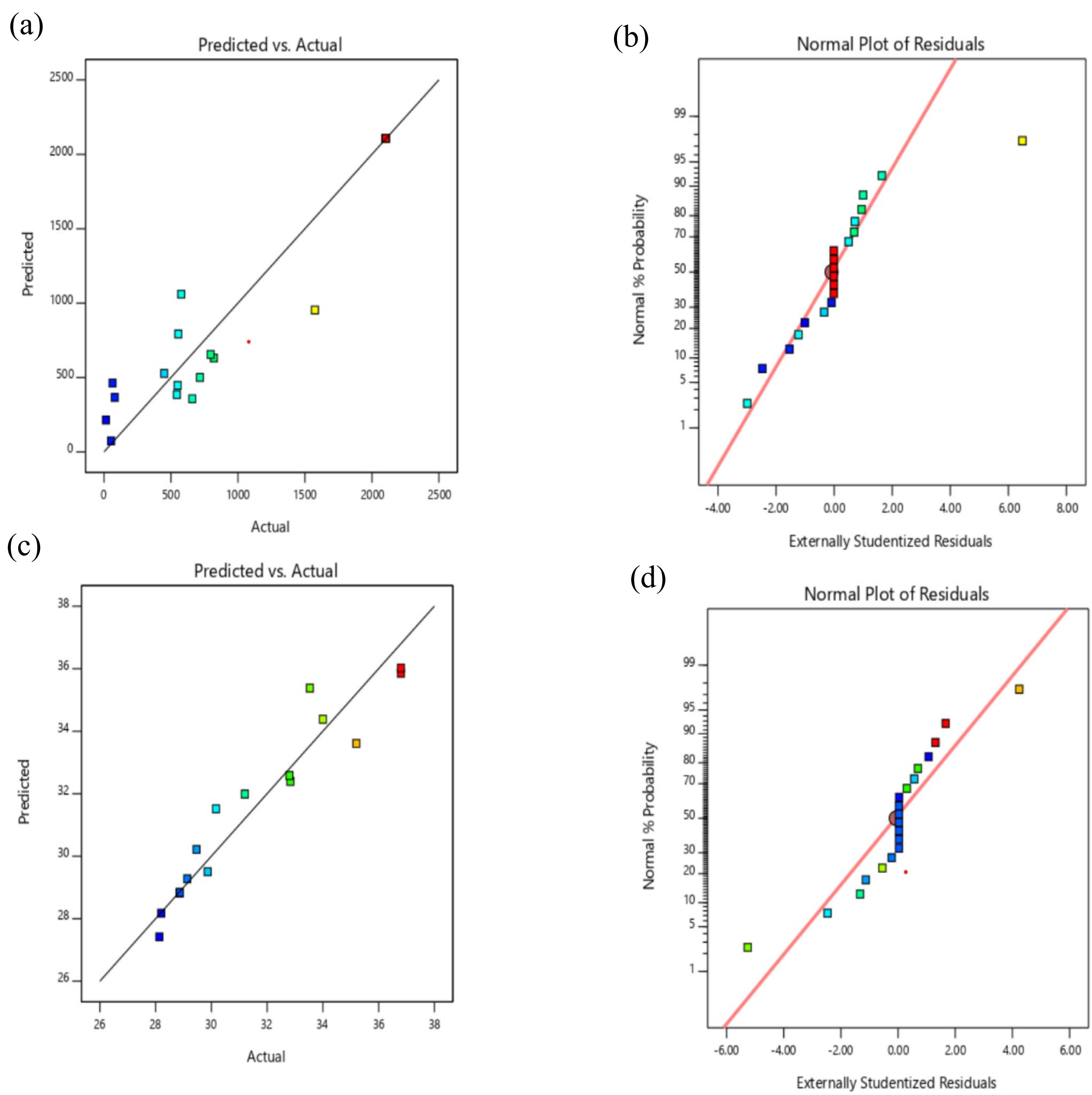

Fig. 4 **a** Predicted vs. actual plot of viscosity; **b** normal plot of residuals of viscosity; **c** predicted vs. actual plot of color; **d** normal plot of residuals of color

discussed indicates that the model is generally reliable despite of the discrepancy. Rocha et al. [42] emphasize that graphical representations are essential for understanding the behavior of residuals in regression models, as they provide insights into the model's fit and help identify potential outliers. This interpretation is consistent with the findings, which discuss how linearity in such plots typically suggests a normal distribution of residuals, although outliers may still be present. Meanwhile for color (Fig. 4(b)), the figure demonstrates a close relationship between the experimental data and predicted line, suggesting an acceptable approximation of the model in estimating the color. This suggests that, in general, the model's predictions for color are very close to the experimental results.

In Fig. 4(b), the residual normal probability plot shows that the data points are closely aligned with the straight line despite exhibiting an S-shaped pattern, suggesting a satisfactory distribution of the residuals. In Fig. 4(d), the normal

Table 4 A Numerical optimization and validation results from RSM

Dabai pulp powder (g)	Glycerol (mL)	CMC (g)	Viscosity (mPa•s)	Color (L*)	Desirability
50	15	12.5	357.72	35.86	0.863
Parameters				Viscosity (mPa•s)	Color (L*)
Predicted value				357.72	35.86
Experimental value				545.32	32.15

Table 5 Comparison of viscosity and color lightness for different samples

Samples	Viscosity (mPa•s)	Color lightness (L*)
Optimized dabai pulp-based marker ink with glycerol	545.32	32.15
Optimized dabai pulp-bsaed marker ink without glycerol	542.73	33.15
Commercial ink	784.03	23.92

probability plot of residuals for color reveals that the data points are generally distributed along a line, though perfect linearity is not fully achieved. However, since the points remain close to the fitted line, this deviation is minimal, and the results can still be considered acceptable. Overall, this alignment reinforces the model's validity and supports its underlying assumptions.

3.4 Optimization and experimental validation

Based on finding in section 3.2, the study suggests that viscosity and color are inversely related. As viscosity increases, the L* tends to decrease while decreasing viscosity results to higher L* value. This correlation highlights the importance of achieving a balance formulation. Thus, by optimizing formulation of CMC and glycerol, it is possible to achieve viscosity and color lightness, ensuring high quality ink. The numerical optimization in this study, using RSM, generated 73 solutions to meet the criteria for optimal viscosity and color characteristics. The solution with the highest desirability score of 0.863 was selected as the optimal parameter set. Table 4 displays the predicted and experimental values for viscosity and color based on these optimal parameters. The best formulation consisted of 50 g of dabai pulp powder, 15 mL of glycerol, and 12.5 g of CMC. The predicted viscosity was 357.72 mPa·s, and the predicted color lightness (L*) was 35.86. Under these optimal conditions, the experimental values were 545.32 mPa•s for viscosity and 32.15 L* for color lightness. The color lightness shows a close match between predicted and experimental results. Although the results of viscosity did not show a good correlation between predicted and experimental values, these results should be interpreted with caution. In future studies, it will be important to consider the physical or theoretical reasons behind these errors. However, this discrepancy may be attributed to the need to verify whether the data falls within the applicable range of the measurement apparatus or theoretical model [43].

3.5 Comparative study of the physico-chemical properties of dabai pulp-based marker ink and commercial marker ink, and the morphological properties of dabai pulp powder

3.5.1 Viscosity and color analysis

Table 5 presents the viscosity and color of the optimized dabai pulp-based marker ink, both with and without glycerol additives, along with a comparison to the commercial ink.

Based on Table 5, the comparison between dabai pulp-based marker ink and commercial marker ink reveals significant differences in both viscosity and color lightness. The optimized dabai pulp-based marker inks, with and without glycerol, exhibit lower viscosities of 545.32 mPa·s and 542.73 mPa·s, respectively, compared to the higher viscosity of the commercial ink at 784.03 mPa·s, indicating that the commercial ink is considerably thicker. This higher viscosity in commercial ink is likely due to the use of more viscous binders and additives designed to regulate ink flow, prevent bleeding, and ensure uniform application across various surfaces. Regarding color lightness, the dabai pulp-based marker inks are lighter, with L* values of 32.15 (with glycerol) and 33.15 (without glycerol), compared to the darker commercial ink, which has an L* value of 23.92. The darker color of the commercial ink is likely due to a higher concentration of pigments and the use of more intense colorants aimed at achieving a vivid and long-lasting mark [44].

The addition of glycerol slightly increases the viscosity and slightly decreases the lightness of the dabai pulp-based marker ink. These results are consistent with those reported by Barreiro et al. [45], who suggest that glycerol not only acts as a humectant but also increases the solution viscosity when added. However, a few studies have reported that the presence of glycerol, a clear liquid, does not affect color attributes [46, 47]. The differences in viscosity and color may stem from variations in the composition and manufacturing processes of the inks.

Our results show that the optimized dabai pulp-based marker ink with glycerol had a viscosity of 545.32 mPa·s, while without glycerol, the viscosity was 542.73 mPa·s, showing only a minimal increase. This observation is consistent with the findings of Hou et al., who reported that glycerol, due to its high polarity and minimal polarity difference from water molecules, has a negligible effect on the viscosity of the mixed system [24]. Though glycerol does not significantly affect viscosity, its role remains important in the extraction of color for this ink. Glycerol acts as extraction solvent, helping to retain moisture and stabilize pigments during extraction, which ensures higher pigment solubility and improved color yield [13]. This is crucial for enhancing the ink's color quality and stability. In contrast, the commercial ink's viscosity of 784.03 mPa•s is much higher, likely due to the use of other additives, synthetic resin, organic pigment, and other alcohols [48], which create stronger molecular interactions and significantly increase viscosity. Having discussed the impact of the viscosity, it is also crucial to understand how pigments affect the color properties of the produced ink. The natural pigments not only enhance or intensify the original color but also ensure consistent coloration, impart color to initially colorless products, and contribute to overall improvements in product quality [49]. This comparison illustrates the variety in viscosity and color across different ink formulations and emphasizes the impact of ingredient selection and their concentrations in achieving the desired rheological properties [50]. However, it is important to consider other ink properties such as pH, surface tension, and its ionic components. Ink pH significantly impacts ink stability where low pH inks improve the pigment stability and adhesion [12]. Meanwhile, higher pH inks help to achieve faster drying and erasability. Other than that, properties such as surface tension helps to identify how well the ink spread. Lower surface tension allows even ink spreading and smooth application. Meanwhile, in Neem gum ink, salts or acids can improve conductivity, affecting ink stability [14]. For berry seed extract ink, ionic components impact adhesion and drying [12]. Conductivity tests reveal if ions are present, helping optimize ink formulation.

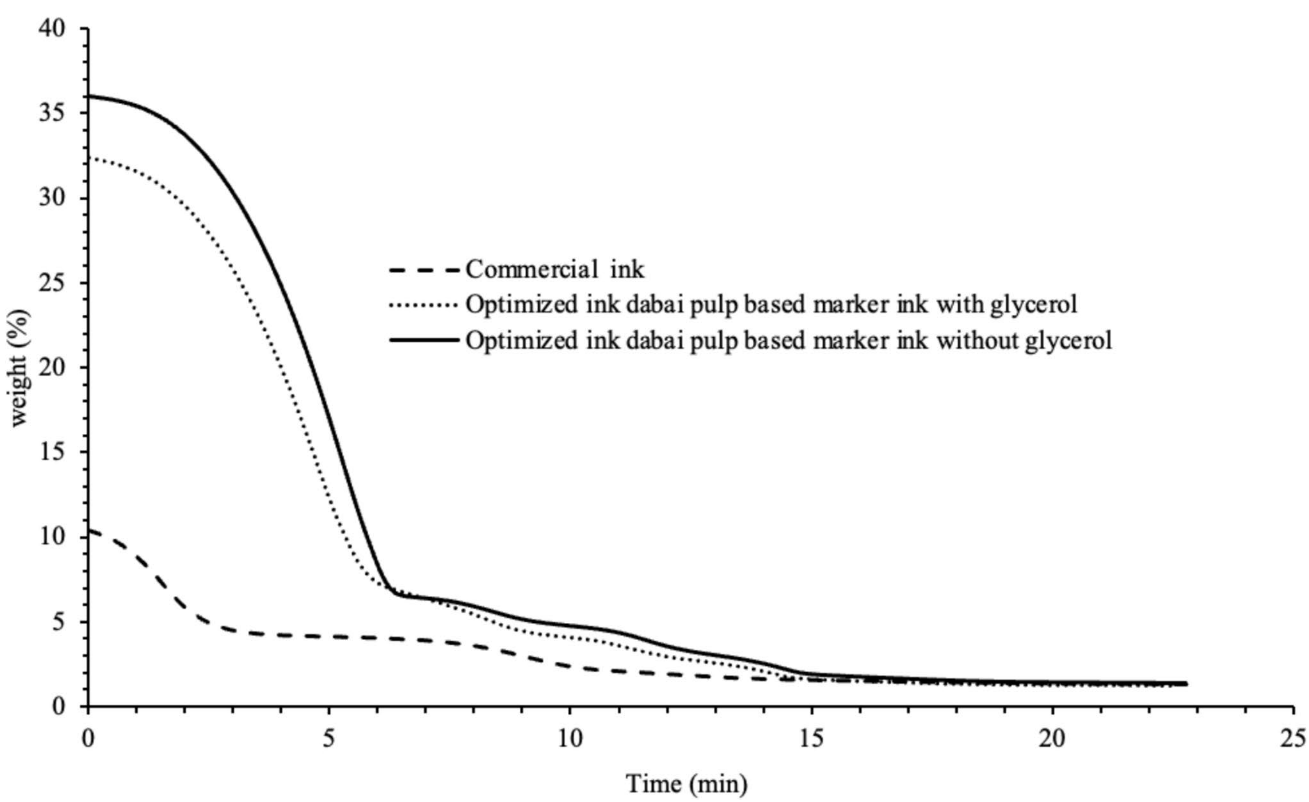

Fig. 5 Weight loss as a function of time for the optimized dabai pulp-based marker ink, with and without glycerol, and the commercial ink

In graphene inks, ions are essential for conductivity, particularly for flexible electronics [18]. These qualities are important especially when it comes to scaling up the ink production process for consistency in pigment stability, adhesion, and drying time. Quality control is essential to maintain performance. The process must also be cost-effective and sustainable, with careful attention to raw material supply and market demand. The availability of raw materials, such as dabai pulp, must be considered. Dabai pulp is currently sourced from seasonal fruits, and its consistent supply may be impacted by seasonal variations and regional availability. As demand in sustainable products grows, dabai pulp-based marker ink offers promise in environmentally friendly industries. However, additional study on its cost and industrial performance is needed for commercial success.

3.5.2 Thermogravimetric analysis (TGA)

The TGA is performed to assess the thermal stability and composition of the ink, especially regarding its degradation behavior under varying temperatures. This is crucial for understanding how the ink behaves under high temperatures and if it will degrade or lose its components at certain temperatures. Figure 5 shows the weight loss (%) of ink samples over time during the TGA test which offers a detailed overview of their thermal behavior. The observed weight loss is attributed to the sorption or desorption of volatiles, as well as decomposition, oxidation, and reduction processes [51]. All three ink samples exhibit a single-stage decomposition pattern.

The TGA analysis shows that the optimized dabai pulp-based marker ink with glycerol (dotted line) exhibited a higher initial weight loss, starting at approximately 32% and decreasing to about 10% within 5 min. In comparison, the optimized dabai pulp-based marker ink without glycerol (solid line) showed a slightly higher initial weight loss of approximately 35%, which dropped sharply to 8% over the same period. The rapid weight loss observed in both samples is due to their higher volatile compound content, leading to rapid evaporation. The discrepancy between the two samples may also be due to the absence of glycerol, which alters molecular interactions and causes phase separation during evaporation [52]. In addition, the presence of glycerol, acting as a plasticizer, reduces the intermolecular strength between adjacent molecules, making them more prone to breaking when exposed to force [53].

The commercial marker ink (dashed line) exhibited the lowest weight loss starting at around 10% and decreasing rapidly to about 2% within 3 min. This may be due to its lower volatile compound concentration and the presence of synthetic resins and additives designed to enhance stability and drying speed. Despite its higher viscosity, the commercial ink showed the fastest drying rate. This result can be attributed to its optimized formulation, where synthetic resins and lower water content allow for rapid solvent evaporation, overriding the effects of high viscosity. Study by Phillips et al. reported that higher-viscosity inks can provide

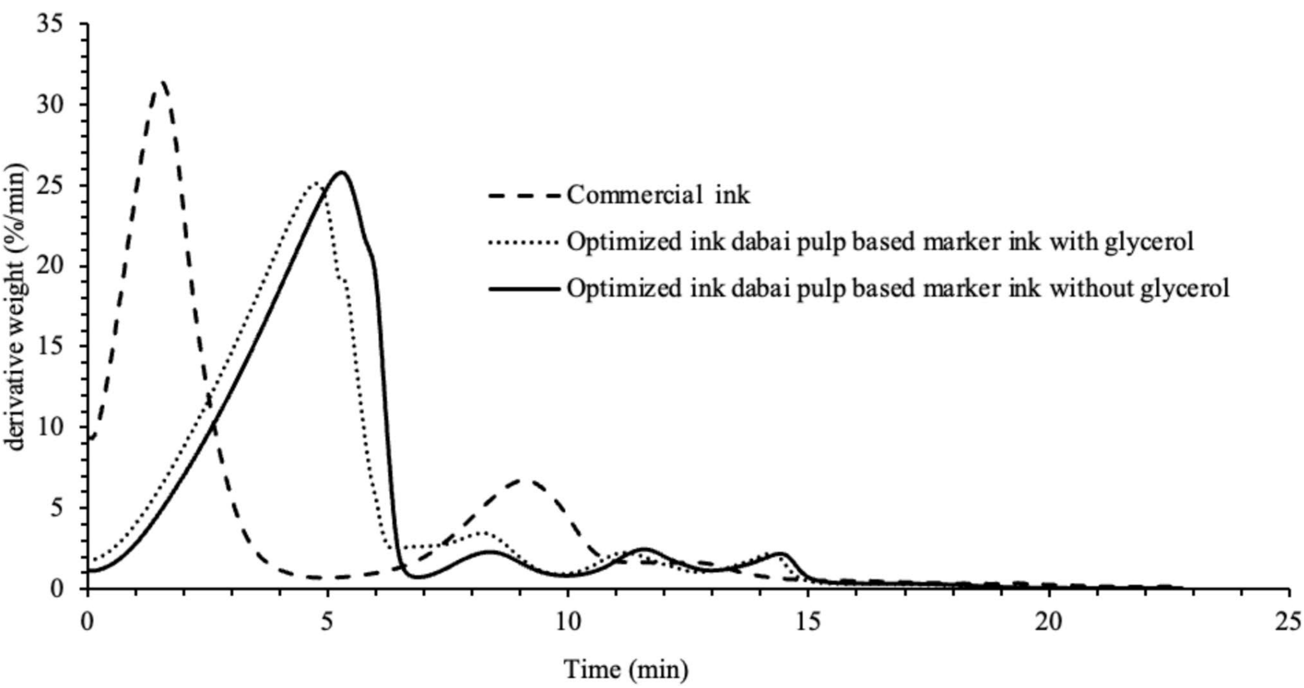

Fig. 6 Derivative weight loss as a function of time for commercial ink, and optimized dabai pulp-based marker ink with and without glycerol

greater color density due to reduced substrate penetration and improved pigment retention [37].

Figure 6 shows the derivative weight loss as a function of time for commercial ink and optimized dabai pulp-based marker ink with and without glycerol.

The commercial ink exhibits a faster drying rate compared to the other two samples. Drying was nearly complete within 3 to 5 min, compared to 7 min for both dabai pulp-based marker inks of lower viscosity. In contrast to theory, the lower viscosity of this sample did not translate to faster drying. Instead, their higher water and volatile content contributed to prolonged drying times by allowing solvent molecules to diffuse more rapidly. Conversely, the high viscosity of the commercial ink did not hinder its drying speed due to its optimized formulation, which includes fast evaporating solvents and binding agents [13]. In many formulations, drying rates are influenced by a combination of factors. Lower viscosity can enhance solvent diffusion, but if the solvent evaporates slowly, has strong binding properties, or if additives hinder evaporation, the drying process may be delayed [54]. On the other hand, reducing viscosity such as by using plasticizers to improve flow may also slow drying if it interferes with solvent evaporation. These interconnected factors highlight the complexity of drying behavior.

While the commercial ink has good performance in drying, both optimized dabai pulp-based marker inks demonstrated superior thermal stability and degradation resistance, which showed their potential for durability in applications. These findings emphasize that while viscosity plays a role in drying behavior, other factors such as solvent composition, resin additives, and volatile content are equally critical. In conclusion, precise control of viscosity and careful consideration of formulation components is important to ensure the quality, stability, and performance of the final product [55].

3.5.3 Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR analysis is applied to assess the marker ink's chemical composition, identify functional groups, and determine how the optimized formulation affects performance and stability [56]. This allows a complete comparison

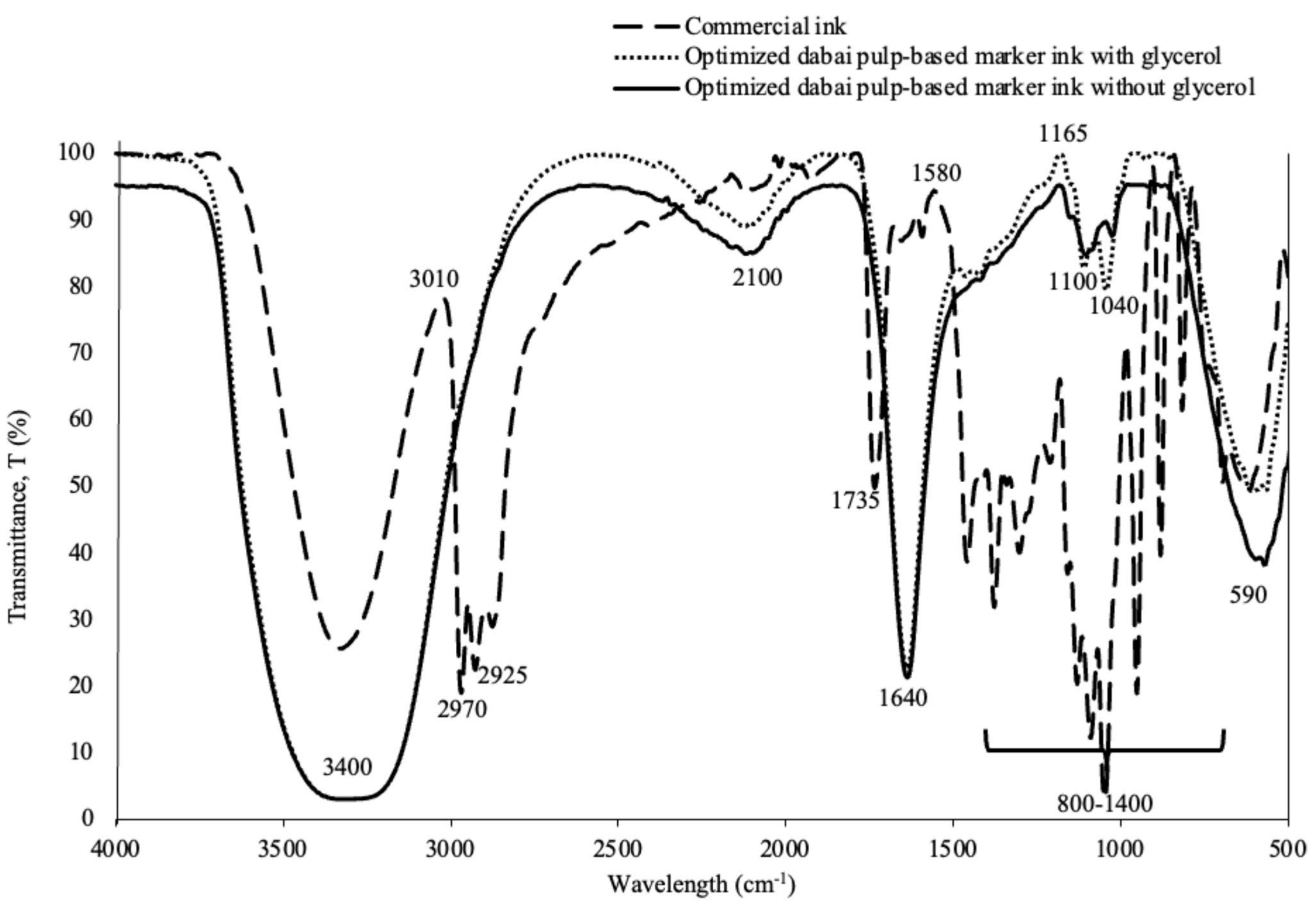

Fig. 7 FTIR spectra of commercial ink and optimized dabai pulp-based marker ink with and without glycerol

between dabai pulp-based ink and commercial inks, revealing the ink's industrial and market potential. The FTIR spectra in Fig. 7 depict transmittance (%) against wavenumber (cm^{-1}) for three ink samples: commercial ink (dashed line), optimized dabai pulp-based marker ink with glycerol (dotted line), and optimized dabai pulp-based marker ink without glycerol (solid line). FTIR analysis in the range of 4000 to 500 cm^{-1} can be used to distinguish between inks based on the presence of solvents, resins, or additives. In this study, most of the ink samples showed peaks at around 2100 cm^{-1}, 1600–1700 cm^{-1}, 1000–1100 cm^{-1}, and 580 cm^{-1}. The similarities and differences in the FTIR spectra were analyzed based on the number of peaks, their positions, and their intensities [57]. There are two main regions in the spectra: the functional group region (above 1500 cm⁻1), which aids in identifying different functional groups, and the fingerprint region (below 1500 cm⁻1), which is used for characterizing the entire molecule [58].

The main finding is that both optimized dabai pulp-based marker inks, with and without glycerol, exhibited a broader intensity band around 3400 cm^{-1} compared to the commercial ink, indicating the presence of O–H groups from the phenolic compounds in their structure [59]. The broad O–H band also partially overlaps with peaks in the 3600 to 3010 cm^{-1} region, which may be attributed to moisture from the ink or the surrounding atmosphere [60]. Additionally, the wavenumbers at 2100 cm^{-1} and 1640 cm^{-1} exhibited a similar pattern of broad intensity when compared to the commercial ink. The peaks at 2100 cm^{-1} referred to C≡C terminal alkyne (monosubstituted) of organic material [61]. The wavenumber at 1640 cm^{-1} is associated with the C = C stretching vibration present in the material [62]. A study by Azman et al. [63] reported that the characteristic bands of CMC appear at 3200–3600 cm^{-1}, 2800–3000 cm^{-1}, and 1000–1300 cm^{-1}, corresponding to the stretching vibrations of hydroxyl groups (–OH), C–H bonds, and ether groups (–O–), respectively.

Few newly emerged absorption peaks were observed in the commercial ink, including 2970 cm^{-1}, 2925 cm^{-1}, 1735 cm^{-1}, and 1550 cm^{-1} that related to the methyl CH asymmetric stretching [64], typical C–H stretch vibrations [65], presence of carboxylic group from acid [66], and a skeletal vibration of dye and the C = C stretch vibration of resin [67], respectively. Both optimized dabai pulp-based marker inks,

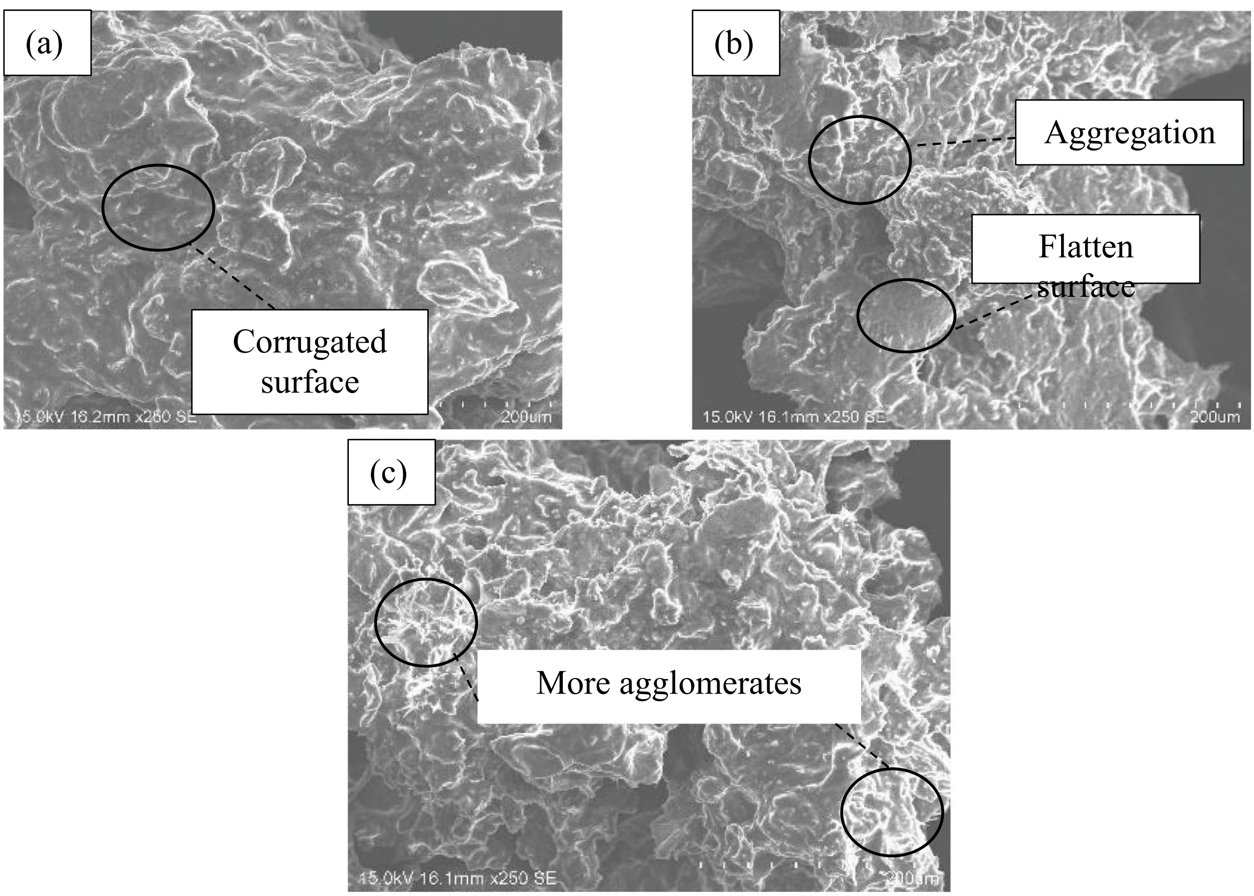

Fig. 8 SEM images: **a** raw dabai powder; **b** optimized dabai pulp-based marker ink powder with glycerol; **c** optimized dabai pulp-based marker ink powder without glycerol

with and without glycerol, exhibit similar chemical compositions at 1100 cm^{-1} and 1040 cm^{-1}, corresponding to the vibrations of aromatic ether and primary alcohol (C–OH) groups, respectively, for both samples [68]. The dabai pulp-based marker ink without glycerol exhibited distinct absorption peaks at 590 cm^{-1}, in contrast to both the dabai pulp-based marker ink with glycerol and the commercial ink. A peak at 590 cm^{-1} is attributed to the O–H out-of-plane bending vibration [69].

3.5.4 Scanning electron microscope (SEM) analysis

Figure 8(a)–(c) illustrates the SEM results showing the structural changes in raw dabai powder, optimized dabai pulp-based marker ink powder with glycerol, and optimized dabai pulp-based marker ink powder without glycerol. The SEM analysis highlights clear differences in the surface structures of these samples. The raw dabai powder, shown in Fig. 8(a), exhibits a bumpy, corrugated surface, which is typical for natural plant powders due to their complex cellular structures. This type of surface is common in natural materials and has been observed in other studies analyzing plant cell structures using SEM [8]. In contrast, the optimized dabai pulp-based marker ink powder with glycerol, shown in Fig. 8(b), has a much smoother and flattened surface due to aggregation. This aggregation occurs because glycerol acts as a stabilizing agent, with its hydroxyl groups facilitating supramolecular arrangements through hydrogen bond formation [70]. The smoothness results from the processing techniques used to enhance the physical properties of the ink, making it more uniform and consistent. Similar findings were reported in a study examining the effects of catalyst ink composition and wet film thickness on fuel cell catalyst layers, which became flatter after chemical processing [71].

Figure 8(c) shows the optimized dabai pulp-based marker ink powder without glycerol, which has an agglomerated surface where the particles clump together. Research on other materials has also shown that the absence of stabilizing agents like glycerol results in agglomerate structures [72]. This agglomeration may hinder pigment dispersion, potentially affecting the ink consistency and application quality. These findings show the importance of additives such as glycerol and CMC in modifying surface structures. The addition of polymers, surfactants, and other substances enhances the stability and dispersion of pigments, helping to achieve the desired color and viscosity while also preventing issues such as clogging during application [73]. The observed differences in surface morphology likely influence the functional properties of the marker ink, with smoother surfaces contributing to better flow and more uniform application.

4 Conclusion

The study successfully optimized the formulation of dabai pulp-based marker ink using RSM. The optimal parameters identified were 50 g of dabai pulp powder, 15 mL of glycerol, and 12.5 g of CMC, resulting in a marker ink with desirable viscosity and color lightness characteristics. The characterization of viscosity and color lightness shows that dabai pulp-based marker inks have lower viscosities, measuring 545.32 mPa·s with glycerol and 542.73 mPa·s without glycerol, compared to the commercial ink's viscosity of 784.03 mPa·s. Additionally, the dabai pulp-based marker inks are lighter in color, with L* values of 32.15 and 33.15 for the ink with and without glycerol, respectively, compared to the darker commercial ink, which has an L* value of 23.92. These findings suggest that dabai pulp-based marker inks produce a lighter color. The TGA results show that commercial inks dry faster, exhibiting lower weight loss due to synthetic resins and additives that enhance drying speed and stability. The faster drying rate of the commercial ink, coupled with its higher viscosity, contributes to its darker color and ensures consistent application, reducing the risk of clumping and uneven distribution. FTIR analysis highlighted the pivotal role of additives such as CMC and glycerol in improving ink formulation. SEM observations revealed that additives and other substances significantly alter the surface structure of dabai pulp powders. In conclusion, using dabai pulp as a primary ingredient in marker ink production supports sustainable practices. This approach reduces dependence on synthetic materials while promoting the use of renewable resources, thereby contributing to environmental conservation.

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Data availability All data generated or analyzed during this study are included in this published article.

Declarations

Ethics approval and consent to participate This article does not contain any studies with human participants or animals performed by any of the authors.

Competing interests The authors declare no competing interests.

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