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# The Effects of Beeswax Additions on the Structural, Thermal, Mechanical and Mass Loss Properties of Soy Wax Blends

*Sharifah Imihezri Syed Shaharuddin*, Mohd Ashhar Mansor*
*Mastura Abdul Rashid, Norhashimah Shaffiar, Zuraida Ahmad*
*Department of Manufacturing and Materials Engineering,*
*Kulliyyah of Engineering, International Islamic University Malaysia,*
*53100, Kuala Lumpur, MALAYSIA*
**shaimihezri@iium.edu.my*

## ABSTRACT

*The typical batik wax compositions used in Malaysia consist of various blends of beeswax, dammar, rosin, paraffin, and microcrystalline wax. This study aims to characterize soy wax/beeswax blends' structural, thermal, strength, and mass loss properties as potential alternative batik wax compositions. The Fourier transform infrared spectroscopy (FTIR) results showed that the additions of beeswax (wt%) into soy wax resulted in possible chemical interaction for the esters (C=O stretching and C-H bending vibrations), hydrocarbons ($CH_2$ scissor formation), and cholestral esters (C=O stretching vibrations). The thermal profile obtained via differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA) respectively revealed an increase in peak melting temperature and reduced thermal stability of the blends with further increments of beeswax. In addition, beeswax enhances the compression strength by 150% but did not have any significant impact on the modulus of the blends. Evaluation of the mass loss test suggested that beeswax in the range of 40 wt% to 60 wt% can be considered as alternative batik resist material due to its moderate leaching tendency. Future works shall be conducted to evaluate the performance of these wax compositions against other batik wax criteria and the feasibility of printing these waxes using an in-house designed batik printer.*

**Keywords:** *Batik Wax; Batik Printer; Thermal; Compression Strength; Leaching*

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## Introduction

The creation of batik textiles involves using various kinds of resist materials to block the diffusion of dye on the fabric and create beautiful batik motifs. In Southeast Asia, batik artisans in Indonesia use wax-based resist materials from beeswax that are blended with shorea javanica (*damar mata kucing*), pine gum (*gondorukem*), wax waste, gum rosin, coconut oil, animal fat, lancing wax, and paraffin [1]-[2]. The ratio of the batik resist materials varies as their batik industry spans across its vast archipelago. The batik artisans located near the coastal areas use different wax blends compared to the batik produced in the cold plateau regions [3]. The batik artisans in Malaysia use wax ingredients that are made up of a blend of beeswax, dammar, rosin, vegetable oil, paraffin, and microcrystalline wax. These waxes are melt-blended at various ratios depending on the desired aesthetic effect. Kudiya et al. [3]-[4] summarised the attributes of a good batik wax as being resistant against alkali and chemicals, durable against hot and cold dye solutions, does not break/crack easily, able to create sharp wax lines, does not create additional staining effects on the fabric, easy to set, non-sticky, not easy to be removed and recyclable.

Wax is a greasy solid that melts readily at low melting points in the range of 40 °C to 75 °C [5] with varying degrees of glossiness, slipperiness, and plasticity [6]. Waxes are categorized as lipids as they are non-polar and only dissolved in organic solvents. The main constituents of wax are hydrocarbons, medium chain length of monoesters-diesters-triesters of fatty acids, long-chain, and free long-chain alcohols, aldehydes, sterols, ketones and ß-diketones, triterpenols, and triterpenic acids [7]-[8]. The use of naturally sourced waxes such as paraffin has raised environmental concerns as they are non-sustainable and non-biodegradable [9]. Hence there is a high demand for "green" waxes. Natural plant-based waxes have risen in popularity due to their abundant supply and low price [10]. Recently, soy oil-based wax has been given attention as a sustainable replacement for paraffin wax [11].

Soy oil is composed of several fatty acids as follows: palmitic acid (10%), stearic acid (4%), oleic acid (18%), linoleic acid (55%), and linolenic acid (13%) [12]. The hydrogenation process at high temperature with nickel catalyst converts the unsaturated fatty acids in the oil into a saturated form. This procedure chemically stabilizes and raises the melting temperature of soy oil resulting in a more solid form of partial or fully hydrogenated soy oil, also known as soy wax [10]. Fully hydrogenated soy wax mainly consists of triglycerides with a high proportion of stearic acid [13]. The use of vegetable oil-based waxes is considered a viable alternative to paraffin as they readily biodegrade compared to paraffin [14]. Despite being hydrophobic, soy wax has a high water uptake [15]. Therefore, the dye colorant may diffuse across the wax lines and leave a staining effect on the batik design. At room temperature, pristine soy wax has a greasy surface texture and is brittle [10]-[11]. Thus, in this study, increments of beeswax are added to improve soy wax properties.

Purified beeswax is processed via various methods such as steam extraction, hot water extraction, and centrifugation [16]. Beeswax consists of multi-component material of at least 284 different compounds [17] and its prominent compound families comprised of 72% fatty acid esters, 13% free acids, and 13% hydrocarbons [18]. Beeswax is a natural water repellant material and has one of the highest water vapor permeability properties attributed to its fatty acids, alcohols, and esters content [19].

A substantial amount of studies have been focused on beeswax [20]. The aromatic hydrocarbon structure of beeswax is said to be responsible for the hydrophobic spreading of beeswax on fabric [21]. The formation of long molecular chains and complex molecules suppresses the formation of stable lamellar structure which contributes to the good pliability nature of beeswax [14]. Kusumawati et al. [1] reported that the presence of a high non-polar functional group in beeswax composition is responsible for its high alkaline resistance. Hu and Yu's [22] study revealed that beeswax's natural structure of hydrocarbon lipid acts as a lubricant that imparts flexibility and viscoelastic properties. Besides Bowen's study [23] on the use of soy wax/beeswax for batik purposes, to our knowledge, information about the thermal, structural, mechanical, and mass loss properties of beeswax and soy wax blends has been lacking.

## Methodology

### Material preparation

The ratios of beeswax (Global Sdn. Bhd., Malaysia) and soy wax (Jargeous Sdn. Bhd., Malaysia) were weighed according to the wt% ratios shown in Table 1. In this study, beeswax was melted first at 70 °C due to its high melt temperature followed by soy wax. Once the soy wax/beeswax blends had completely dissolved, the prepared batik wax blends were then left to solidify for 48 hours before being re-melted for ensuing tests.

Table 1: Sample codes for the soy wax/beeswax blends

| Sample code | Beeswax (wt%) | Soy wax (wt%) |
|---|---|---|
| Neat beeswax | 100 | - |
| Soy20bees80 | 80 | 20 |
| Soy40bees60 | 60 | 40 |
| Soy60bees40 | 40 | 60 |
| Soy80bees20 | 20 | 80 |
| Neat soy wax | - | 100 |

## Fourier Transform Infrared Spectroscopy (FTIR)

Identification of the functional group of soy wax/beeswax blends was obtained by using Tensor 27 FTIR Spectrometer (Bruker, USA). The spectra were collected within 4000-6000 $cm^{-1}$ regions with a resolution of 4 $cm^{-1}$ and 16 scans.

## Thermal characterizations

The thermograms of beeswax and soy/beeswax blends were obtained using a Sapphire Differential Scanning Calorimetry (Perkin Elmer Instrument, USA). About 5 mg of the sample was sealed in an aluminum pan and heated from 30 ℃ to 200 ℃ at a rate of 10 ℃/min and a constant flow rate of 50 ml/min nitrogen gas. Two readings were taken for each sample.

The thermogravimetric analysis (TGA) of the soy wax/beeswax samples was performed using a Simultaneous Thermal Analyzer (STA7300) (Hitachi High Technologies, USA). The thermal profile of the samples was determined using approximately 15 mg to 20 mg under an air atmosphere at a heating rate of 10 ℃/min, from 40 ℃ to 450 ℃. The initial degradation temperature ($T_i$) and the temperature for the final degradation ($T_f$) were obtained based on extrapolated onset data.

## Compression test

The soy wax/beeswax compositions were remelted and cast into a coated cylindrical acrylonitrile butadiene styrene (ABS) mold with a 30 mm diameter. Upon solidification, the cylindrical soy wax/beeswax samples were removed from the mold and cut into 40 mm lengths as per the compression test for beeswax by Morgan et. al [17]. Five samples (n = 5) for each composition were prepared for compression test using Shimadzu Universal Tensile Machine (AGS-5K NXD). The test was performed at a crosshead speed of 1 mm/min and a load cell of 5 kN at an ambient temperature of 27 °C.

## Mass loss test

A 3 cm x 3 cm inner square was sketched on each 8 cm x 8 cm square cotton fabric. The wax blends were re-melted, and a glass syringe was used to apply a consistent wax volume of 1 ml onto the 3 cm x 3 cm inner square. Six samples were prepared for each composition. The mass of the wax-covered cotton fabric was weighed to obtain its initial mass ($M_i$). Each waxed fabric sample was then immersed in 1000 ml of 100 °C water for 5 and 15 minutes. The sample fabric was then removed and dried for 24 hours. The final mass ($M_f$) was measured for each sample. The mass loss (%) was calculated according to Equation (1).

$$\text{Mass loss \%} = \frac{M_i - M_f}{M_i} \times 100\% \tag{1}$$

## Results and Discussions

### Fourier Transform Infrared Spectroscopy (FTIR)

Figure 1 shows the absorption of infrared light at different wavelengths of the studied soy wax/beeswax blends. The FTIR spectra of soy wax showed the typical peaks at 718 $cm^{-1}$ and 1176 $cm^{-1}$ and were assigned as C-C group vibrating and C = O stretching respectively. Two distinct peaks were also detected at 1741 $cm^{-1}$ and 2917 $cm^{-1}$ which arose from C = O stretching and $CH_2$ asymmetric stretching respectively. The FTIR peaks for soy wax in this study are consistent with the available data reported in the literature [22], [24]-[25].

Distinctive compounds or ′fingerprints′ for beeswax occur in the spectral region of 1800 $cm^{-1}$ and 800 $cm^{-1}$. These high absorption bands correspond to the monoesters and carboxyl group of free fatty acids [26]-[27]. FTIR peaks at absorbance intensities of 1172 $cm^{-1}$, 1463 $cm^{-1}$, and 1736 $cm^{-1}$ were also noted and are consistent with published values [5], [27]. It can also be confirmed that the wax used in this study is indeed beeswax rather than other hydrocarbon compounds due to the strong peak intensity at 1736 $cm^{-1}$ which indicates the formation of an ester bond between fatty acid and cholesterol’s hydroxyl group [28]. The sharp peak at 2849 $cm^{-1}$ corresponds to aliphatic hydrocarbon chains for $CH_2$ symmetric stretching vibrations and the peak at 2918 $cm^{-1}$ is due to $CH_2$ asymmetric stretching vibrations.

Table 2 tabulates the wavenumber and assigned functional groups for the FTIR absorption. It was observed that further increments of beeswax into soy wax resulted in an apparent peak position shift of the beeswax’s ′fingerprints′ compound towards higher wavenumber suggesting chemical interaction for the esters (C = O stretching and C-H bending vibrations), hydrocarbons ($CH_2$ scissor formation), and cholestral esters (C = O stretching vibrations).

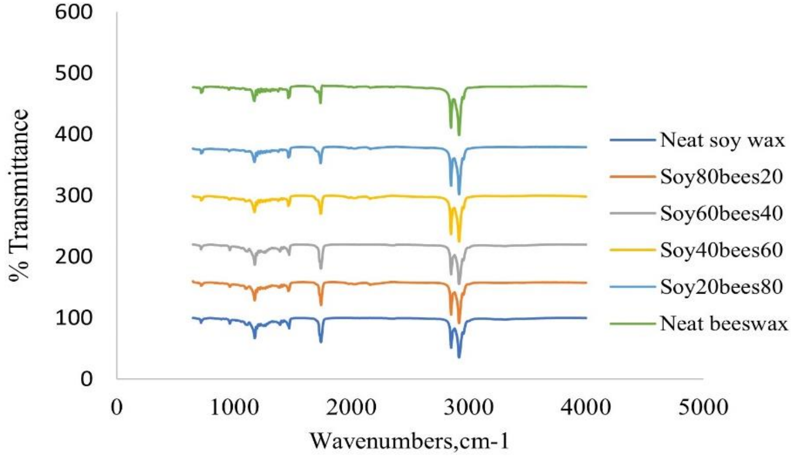

Figure 1: FTIR spectra of soy wax/beeswax blends

Table 2: Assigned wavenumber and vibrating group for the soy wax/beeswax blends used in this study

| Sample code wavenumber | | | | | | Vibrating group |
|---|---|---|---|---|---|---|
| Neat soy wax | Soy 80 bees 20 | Soy **60** bees **40** | Soy **4**0 bees **6**0 | Soy **2**0 bees **8**0 | Neat beeswax | |
| 718 | 720 | 720 | 720 | 720 | 720 | $CH_2$ rocking mode (hydrocarbons - alkenes) |
| 1176 | 1175 | 1175 | 1175 | 1174 | 1172 | C=O stretching and C-H bending vibrations (ester) |
| 1470 | 1465 | 1463 | 1463 | 1463 | 1463 | $CH_2$ scissor formation (hydrocarbons -alkanes) |
| 1741 | 1742 | 1741 | 1739 | 1738 | 1736 | C=O stretching vibrations (cholesterol esters) |
| 2850 | 2850 | 2849 | 2849 | 2850 | 2849 | $CH_2$ symmetric stretching vibrations (aliphatic hydrocarbons) |
| 2917 | 2918 | 2918 | 2918 | 2918 | 2918 | $CH_2$ asymmetric stretching vibrations (aliphatic hydrocarbons - alkanes) |

## Thermal properties

Figure 2 shows the DSC thermograms of the soy wax/beeswax blends used in this study. The glass transition temperature for neat soy wax and neat beeswax was hardly discernable from the DSC curve suggesting that these waxes consist of heavily crosslinked molecular chains that eventually disrupt the ease of cooperative segmental mobility. Soy wax's thermogram showed a single smooth melting peak at 57 °C, which is possibly due to the heat absorbed by esters and partial acyl glyceride with long and saturated acyl chains [10]. It is also concluded from the thermogram that the soy wax used in this study is in the fully hydrogenated form since the melting peak occurred in the range of 53 °C-63 °C [11]. In comparison to soy wax, beeswax exhibited higher melting temperatures as it is composed of mixed esters with high molecular weights and complicated structures [21]. Beeswax's melting peak in the range between 40 °C to 60 °C is associated with the heat absorbed by free acids and hydrocarbons, whilst a second larger and well-defined peak detected at 72 °C is due to the heat absorbed by the dominant fatty acid and wax ester component [18], [31]. Beeswax also exhibited a broader melting peak region compared to soy wax. Therefore it is anticipated that melted beeswax can spread easily and penetrates better through the cotton fabric before becoming completely solidified [26]. Further additions of beeswax (wt%) into soy wax increased the melting temperature of the blends.

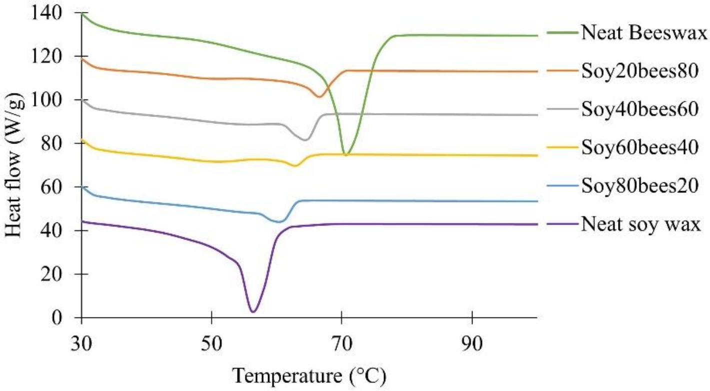

Figure 2: DSC thermogram of the soy wax/beeswax blends

Figure 3 shows the weight-loss (%) trends of the soy wax/beeswax blends as a function of temperature. Each composition exhibited an initial plateau region of constant mass followed by a single-stage decomposition. Neat soy wax is the most thermally stable material compared to other waxes in this study as its thermal degradation region occurred between 376 °C and 450 °C. Neat beeswax experienced thermal degradation starting at 304 °C. As it reached

400 °C, neat beeswax experienced 70% weight loss due to the degradation of most of its polar and non-polar bonds in its hydrocarbon and esters [32]-[33]. Beyond 430 °C further decomposition took place for the beeswax's long-chain molecules [26] resulting in more than 90% weight loss Further addition of beeswax (wt%) into soy wax resulted in a reduction in thermal stability of the soy wax/beeswax blends from 376 ℃ to 328 ℃. It was also noted that all studied waxes underwent complete pyrolysis with almost no remaining residues beyond 480 °C.

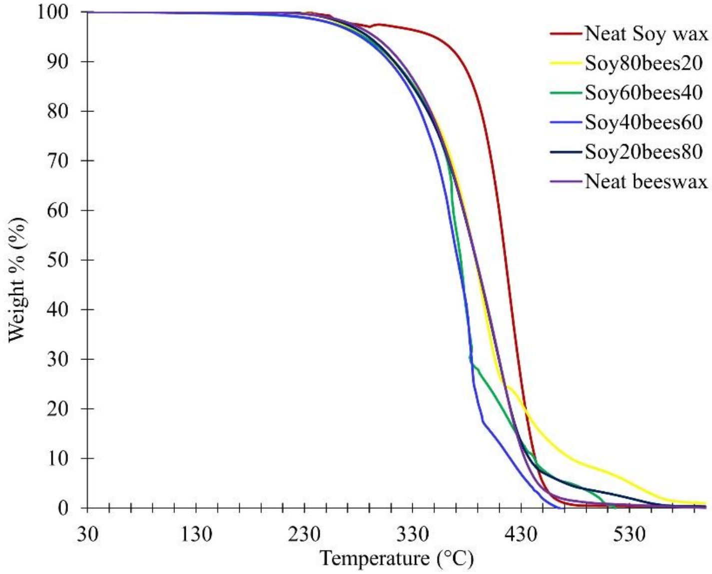

Figure 3: Thermogravimetric analysis of soy wax/beeswax blends

## Mechanical properties

The stress-strain profile and the average ultimate strength and modulus for the soy wax/beeswax blends are shown in Figure 4 and Figure 5, respectively. The compressive stress-strain profile shows that the waxes behaved in a ductile manner as their stress values gradually decreased beyond their well-defined yield point of ultimate strength. In comparison to soy wax, the compression test results indicated that neat beeswax had higher ultimate strength attributed to the presence of fatty acid components in its chemical structure [32]. The ultimate strength of neat beeswax at 0.8 MPa is slightly lower than the value reported by Morgan et al. [17]. Their study reported a strength value of 1.0 MPa since their test was conducted at a lower temperature of 24 °C. Increments of beeswax additions up to 80 wt% into soy wax resulted in a significant increase in compression strength of the blends by approximately 150%.

However, the additions of beeswax did not impart any improvements on the modulus of the blends due to beeswax's inherently pliable nature [14]. Images of the samples under compression are shown in Figure 6. It was observed that neat soy wax responded to the compression load with a higher number of cracks compared to beeswax, which reflects the brittle and lack of cohesiveness of soy wax.

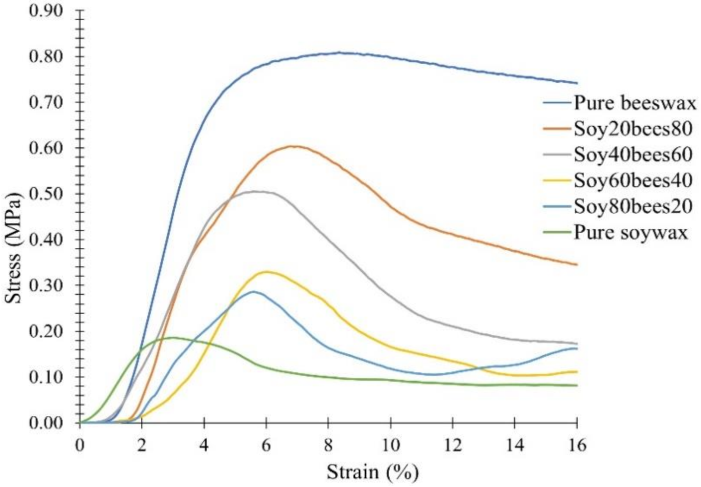

Figure 4: Stress-strain curves of soy wax/beeswax blends via compression test

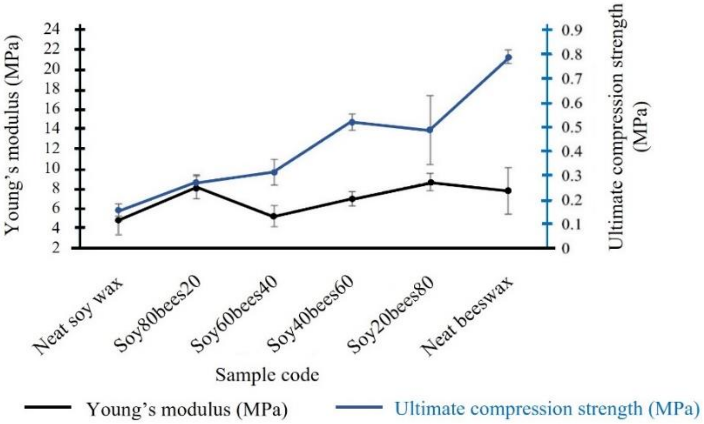

Figure 5: The ultimate compression strength and Young's modulus of soy wax/beeswax blends via compression test

Figure 6: The soy wax/beeswax specimens under compression load

## Mass loss properties

Figure 7 shows the blends' mass loss (%) upon immersion in 100 °C water for 5 minutes and 15 minutes. Beeswax and soy wax are superhydrophobic materials [13], [33]-[34]. However, at high immersion temperatures, these materials' superhydrophobic properties denature which enables the leaching of the waxes into the water. It was observed that soy wax recorded the highest mass loss of 33% and 39% for the immersion period of 5 minutes and 15 minutes respectively. DSC thermogram showed that soy wax has a low melt temperature which may have made it easier for the soy wax to disintegrate and leach into the immersion water. Bowen’s [23] study also observed that soy wax used for batik applications can be easily dissolved in water and can be removed using the hot iron technique. In comparison, beeswax showed the lowest mass loss at 5% and 12% for the immersion period of 5 minutes and 15 minutes respectively.

It was evident from the mass loss (%) trends that the initial addition of 20 wt% of beeswax for Soy80bees20 composition resulted in a marked reduction of mass loss (%) of the wax blend for both immersion periods. Further beeswax additions (wt %) led to a decreasing mass loss (%) trend. The result suggests that beeswax provides a better barrier against water contact with the surface of the wax blends which led to a reduced leachability into the immersion water. However, it was also noted that further additions of beeswax at 60 wt% and 80 wt% did not show an appreciable mass loss difference for the 15 minutes immersion period compared to the 5 minutes immersion period. The leaching of material into the immersion medium behaves in a nonlinear manner [35]. During the 5 minutes immersion period, the amount of mass loss (%) is attributed to the initial leaching of the waxes due to surface contact with water. As the immersion period was increased to 15 minutes, the leaching of the soy wax/beeswax blends eventually slowed down and reached a limiting value possibly as a consequence of intraparticle water diffusion into the bulk of the solid waxes that are attached to the cotton fabric.

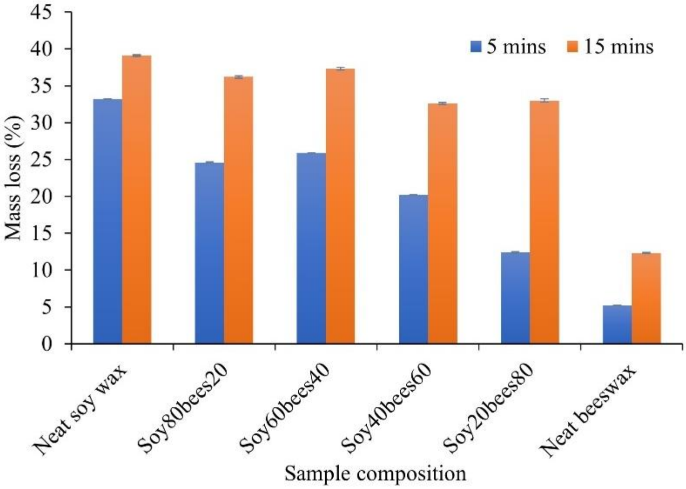

Figure 7: Mass loss (%) for soy wax/beeswax blends at 100 °C water after 5 minutes and 15 minutes

In batik production, the leaching tendency of the waxes is an important parameter that affects its overall process. Batik waxes with a low leaching performance increase the time and energy consumption during the wax removal phase. On the other hand, wax compositions with a high leaching tendency facilitate the wax removal phase but may not be robust enough to be used in multiple dyeing processes which limits the creative batik process. Wax compositions with beeswax in the range of 40 wt% to 60 wt% provided moderate leaching performance, therefore can be considered as alternative batik wax resist materials. Further studies are currently being conducted to investigate whether these compositions can impart other batik wax qualities such as the ability to impart ‘crackle’, a batik trademark effect [36], and continuous wax lines via a recently developed batik printer.

## Conclusions

Worldwide enforcement for ‘greener’ products has instigated this study on the characterization of alternative sustainable and biodegradable resist material consisting of soy wax/beeswax blends. The effect of blending beeswax into soy wax was noted with a chemical interaction for the FTIR ‘fingerprint’ region for beeswax. The $T_g$ for soy wax and beeswax was hardly discernable from the DSC curve suggesting that these waxes consist of heavily crosslinked structures that eventually disrupt the ease of segmental mobility. The study also showed a decreasing trend in thermal stability as measured from the initial temperature ($T_i$) of the TGA profile with subsequent beeswax additions. The results obtained suggest that the incorporation of beeswax is effective in improving the strength as well as leaching properties of the soy wax blends which shows the promising application as batik resist material.

## Contribution of Authors

The authors confirm the equal contribution in each part of this work. All authors reviewed and approved the final version of this work.

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## Conflict of Interests

All authors declare that they have no conflicts of interest

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