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ARTICLE

Unveiling the Signature of Halal Leather: A Comparative Study of Surface Morphology, Functional Groups, and Thermal Characteristics

Muh Wahyu Syabani[1]*, Iswahyuni[2], Warmiati[3], Kutut Aji Prayitno[4], Henny Saraswati[5], Rahmandhika Firdauzha Hary Hernandha[6]

Abstract: *The halal certification of products holds significant importance for Muslim consumers, necessitating the development of reliable techniques for identifying leather products made from raw materials. This study employed rapid and accurate analytical methods to distinguish between cowhide, pigskin, and artificial leather. A combination of scanning electron microscope (SEM), Fourier transform infrared spectroscopy (FTIR), and differential scanning calorimetry (DSC) was used to assess the variations in collagen fiber structures and thermal stability among the leather samples. The findings revealed that morphological surface analysis, including grain patterns and pores, facilitated swift differentiation between different leather types. Pigskins exhibit three-hole patterns on their morphological surface compared to cowhide, with random pores and tighter grain patterns, whereas artificial leather lacks natural grain patterns and pores altogether. While FTIR spectra exhibited similarities between cowhide and pigskin leathers, variations in vibration intensity enabled effective discrimination. Artificial leather, particularly PVC-based materials, displayed distinct spectra, allowing FTIR spectroscopy to effectively discern between halal and non-halal leather. Cowhide possesses strong and sharp vibration at wavenumber 1736, 1277, and 817 cm^{-1} compared to pigskin, which has stronger vibration at 1534 cm^{-1}. Meanwhile, PVC-based artificial leather exhibited stretching at 1723 and 744 cm^{-1} wavenumbers. DSC analysis proved valuable in differentiating between genuine and artificial leather based on unique peaks and thermal behavior. These three techniques provide reliable means to determine the raw material origins of leather products.*

Keywords: *DSC, FTIR, halal, leather, SEM*

e-mail: mwsyabani@atk.ac.id*[1], iswahyuni@atk.ac.id[2], warmiati@atk.ac.id[3], kututaji@atk.ac.id[4], henny.saraswati@uinbanten.ac.id[5], hernandha.en07@nycu.edu.tw[6]
*Corresponding Author

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[1,2,3,4]Department of Rubber and Plastic Processing Technology, Politeknik ATK Yogyakarta, Jl. Prof. Dr. Wirjono Prodjodikoro, Yogyakarta, 55188, Indonesia. [5]Faculty of Islamic Economics and Business, Universitas Islam Negeri Sultan Maulana Hasanuddin, Jl. Raya Syeikh Nawawi Albantaniy, Banten, 42171, Indonesia. [6]Department of Materials Science and Engineering, College of Engineering, National Yang Ming Chiao Tung University, No. 1001 University Road, Hsinchu, 30010, Taiwan.

1. Introduction

Tannery has been an essential industry since ancient times (Liu et al., 2016). Chemically modifying animal skin results in more robust and more flexible materials, which can prevent further damage (Miao et al., 2021). These materials have been widely used in the fashion industry, footwear, automotive, technical materials, and other applications (Hermiyati et al., 2017; Meyer et al., 2021). Due to their extensive use in everyday life, the demand for leather products continues to increase (Izuchi et al., 2016). Leather can be produced from various raw materials. Material from larger animals, such as cattle, horses, and buffaloes, are called hides. On the other hand, the skin usually refers to smaller animal skins like goats, pigs, dogs, and fish.

Transparency in leather products is necessary to maintain their quality and value. Therefore, it is crucial to verify the origin of the raw leather material for such products (Izuchi et al., 2016). People worldwide have diverse beliefs and lifestyles related to the use of leather. Hindus consider cows sacred animals and are sensitive to issues related to leather derived from this animal (Tejani, 2019). However, according to Islamic beliefs, pig leather is considered non-halal, even if it has undergone tanning. The halal status of products is of great importance to Muslim consumers, reflecting their strong emphasis on adherence to Islamic principles (Fajriati et al., 2021; Hermanto et al., 2022; Kashim et al., 2023). This situation requires special attention to prevent consumer anxiety due to these sensitive issues. Furthermore, Indonesia possesses excellent potential to flourish and become a global leader in the halal industry (Yuniastuti & Pratama, 2023).

Recently, some manufacturers have used pig leather as a primary product component (Mirghani et al., 2012). In certain countries, such as Japan and Spain, labeling indicating the origin of the raw materials has been implemented (Bañón et al., 2021; Izuchi et al., 2016). However, non-halal products often do not have clear labeling. Thus, consumers are often unaware of the origin of the raw materials used in the leather products they purchase. Furthermore, there has been a significant advancement in the development of artificial leather, allowing it to closely resemble the characteristics of genuine leather (Meyer et al., 2021; Pullawan, 2016). As a result, it has become essential to differentiate between these two types of leather through species identification (Ma et al., 2019). Consequently, there is a potential for consumers to unknowingly use leather products that contain materials they do not desire.

There is already a standard for identifying leather from other materials using ISO 17131:2020, but this standard is not explicitly intended for specific types of leather. The neural network approach can also be used to identify and classify leather (Kwak et al., 2000). However, the mentioned study only discusses the classification of leather-based on its defects. Surface analysis and spectral values of leather can be examined for identification purposes using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy (SEM) techniques (Carsote et al., 2021; Mirghani et al., 2012). The identification of vegetable tanning agents on ancient book covers made of leather can be carried out using FTIR (Liu et al., 2016; Puică et al., 2006; Sebestyén et al., 2022). Liquid Chromatography/Mass Spectrometry (LC/MS) methods are also being developed to identify amino acids in various types of leather (Gao et al., 2021; Izuchi et al., 2016).

Based on the above discussion, it is necessary to continue developing various techniques to identify leather products from different raw materials, especially those sensitive to consumers, such as cow and pig leather. Whereas differential scanning calorimetry (DSC) has been successfully applied in thermal analysis for polymers and pharmaceuticals, its potential for leather identification remains unexplored. Therefore, this study aims to identify leather products made from cows, pigs, and other types of leather, including artificial leather, using surface morphology, functional groups, and thermal analysis. Moreover, DSC is expected to provide an alternative approach for identifying leather product raw materials.

2. Materials and Methods

The materials used in this research are cow leather, pig leather, and polyvinyl chloride (PVC) based artificial leather. These materials were obtained from local distributors in Indonesia and the Polytechnic of ATK Yogyakarta.

2.1. Surface Morphology

The surface appearance of the leather samples is determined through visual observation using a digital microscope (Motic) and scanning electron microscope (miniSEM SNE 4500M). The leather samples were cut into 1 cm × 1 cm sizes and placed on a sample holder with carbon tape. Subsequently, a conductive material was applied as a coating to the samples. The coated samples were subjected to analysis using SEM at a magnification level of 35×.

2.2. Functional Groups Analysis

FTIR spectroscopy (PerkinElmer Frontier UATR) was employed to acquire FTIR spectra for the samples. The leather samples were prepared in 2 cm × 2 cm dimensions and positioned on a sample holder for analysis within the wavelength range of 4000–600 cm^{-1} under ambient conditions.

2.3. Thermal Analysis

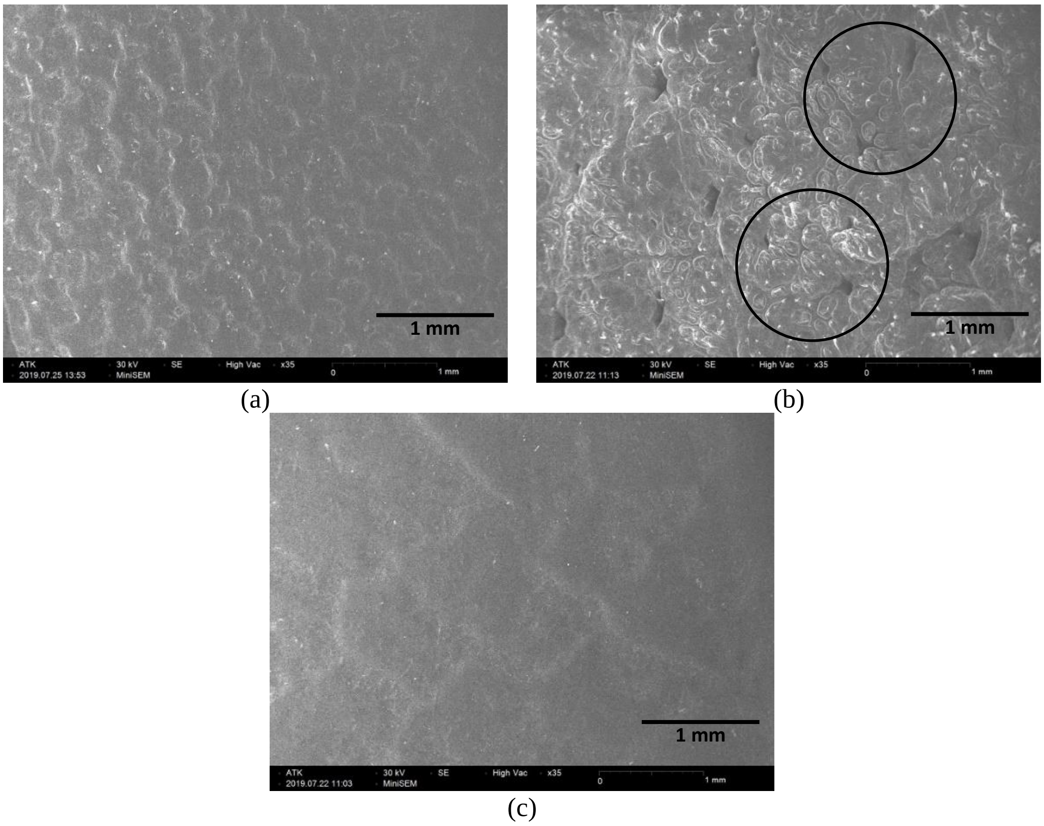
The temperature influences on the leather are investigated by conducting thermal analysis using a differential scanning calorimeter (PerkinElmer DSC4000). The leather samples, weighing approximately 5–10 mg, are placed in alum standard pans and then compressed using a sample crimper. The DSC operates within a temperature range of 10–445°C, with a heating rate of 10°C/minute, and in a nitrogen atmosphere at a 20 mL.min^{-1} flow rate.

3. Results and Discussion

3.1. Surface Morphology

The morphological surface analysis involves visually examining and inspecting the leather structure using magnification instruments such as digital and scanning electron microscopes. Differences in grain patterns, pores, or the presence of scales on the leather surface can aid in distinguishing between different types of leather (Ebsen et al., 2019). The resulting images are presented in Figure 1.

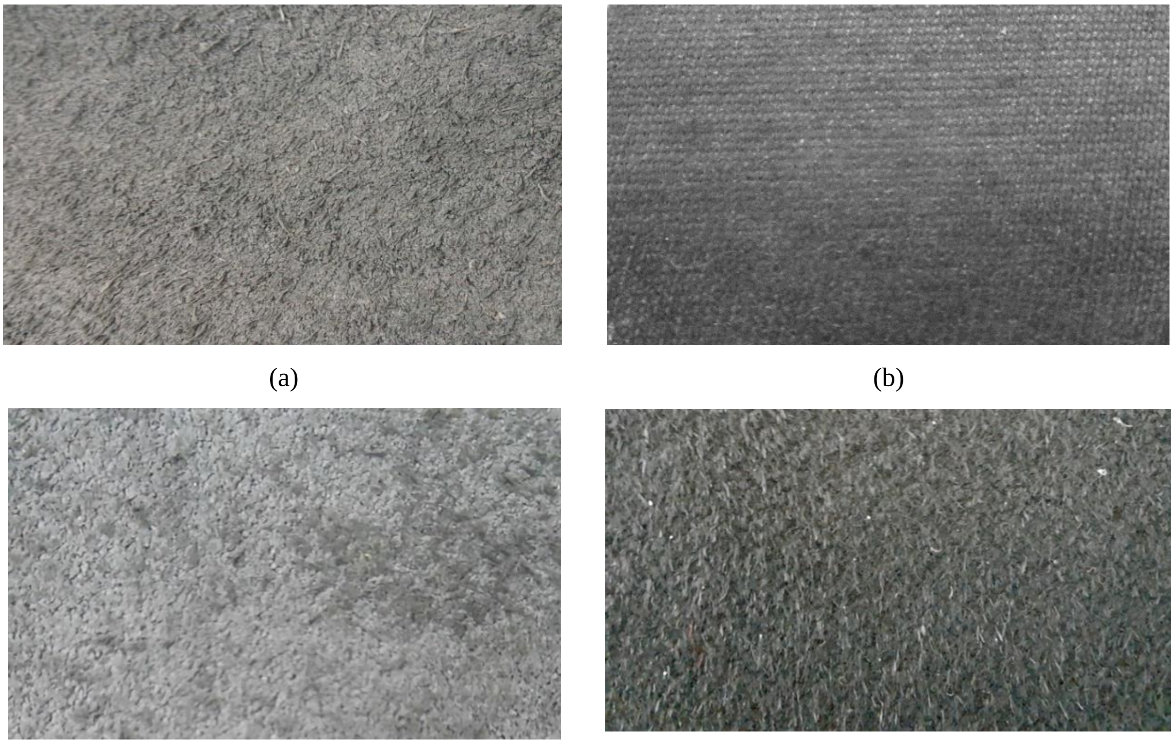
Pig leather has the characteristic of being strong but stiff (Mirghani et al., 2012). The distinctive feature of pig leather is the grain pattern generated by hair growth, which includes groups of three holes. These holes remain present during the dehairing process, both on the grain and flesh sides. In Figure 1(b), the three-hole pattern of pig leather can be seen clearly. Cow leather possesses random pore and tight grain pattern as shown in Figure 1(a). A similar random pattern can also be found to buffalo and sheep leather (Varghese et al., 2022). In comparison, from Figure 1(c) above, in artificial leather products, the surface is relatively smoother than in genuine leather. However, significant progress has been made in developing artificial leather products, allowing for printing surface textures, and including breathing holes to mimic natural leather texture closely (Meyer et al., 2021). One distinguishing characteristic of genuine leather is the presence of pores, which provides ventilation for the wearer's skin leather products (Fathima et al., 2010). Pores in artificial leather can confuse potential buyers in differentiating it from authentic leather (Pullawan, 2016). However, we can still differentiate the distinction between the inner side of the leather, as illustrated in Figure 2.



(a) (b)

(c)

Figure 1. SEM imaging with 35× zoom for cow leather (a), pig leather (b), and PVC (c).



(a) (b)

(c) (d)

Figure 2. Backside imaging results in pig leather (a), artificial leather (b), cow leather (c), and goat leather (d).

The inner surface of both genuine and artificial leather is displayed in Figure 2 above. The distinguishing factor between these types of leather is the presence of pores that penetrate from the grain side to the flesh side in genuine leather. On the other hand, in the case of artificial leather, the pores are often not visible on the inner side. It is because artificial leather production typically involves multiple layers of polymer, and the inner part is reinforced with a fabric layer (Gilon et al., 2023; Syabani et al., 2022). A clear illustration of the layer comparison between genuine and artificial leather is shown in Figure 3.

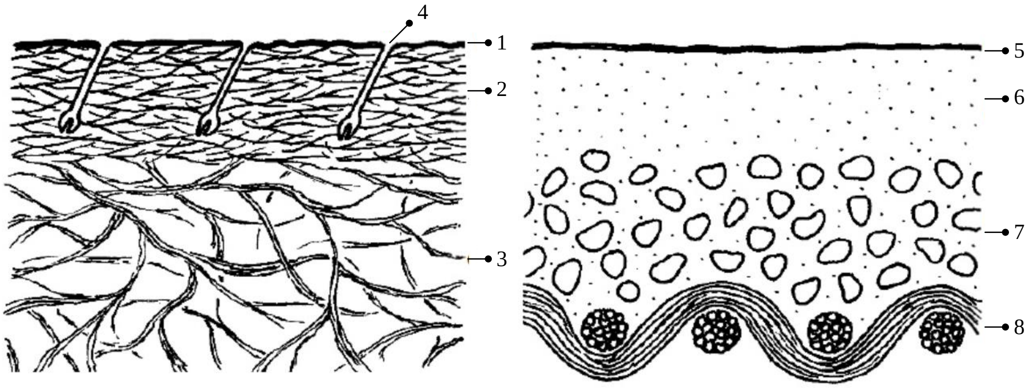


Figure 3. The layer structure of genuine leather (a) and artificial leather (b). Leather retains the histological structure of the skin, comprising the grain membrane (1), papillary layer (2), reticular layer (3), and hair channels (4). On the other hand, artificial leather consists of a topcoat (5), a compact layer (6), a foamed middle layer (7), and a fabric support layer (8) (Meyer et al., 2021).

Based on the above description, there is a clear distinction between pig leather and cow leather, primarily attributed to a three-hole pattern on pig leather appearance. In the Figure 3(a), the cross-sectional views of genuine leather displayed densely packed collagen fibers along with a thin grain layer (Gao et al., 2021). While the inner reticular layer structure is more open thus less hard than the grain (Bañón et al., 2021). In contrast, as shown in Figure 3(b), although it can imitated the structure and printed with pores that resemble genuine leather, artificial leather still can be identified by its relatively uniform formation of breathing holes and the utilization of a supporting fabric on the backside (Ambroziak & Kłosowski, 2014; Duo et al., 2019).

3.2. Functional Groups

Fourier Transform Infrared Spectroscopy (FTIR) is a non-destructive analytical technique used to examine chemical functionalities within materials by detecting the characteristic vibrations of chemical bonds (Pullawan, 2016). This method relies on a material's absorption of infrared radiation, enabling the identification and analysis of its chemical composition. The advantage of FTIR spectroscopy is its ability to eliminate the need for time-consuming and chemical-intensive standards, thus giving rapid, consistent, and reproducible analytical techniques (Mirghani et al., 2012). This study's primary focus is identifying non-halal pigskin leather using reliable and efficient FTIR analysis, representing a novel analytical approach. The research involves the analysis of three different types of leather, namely cowhide, pigskin, and artificial leather, which are then compared to the spectra of natural leather used for authentication purposes. In FTIR analysis, several leather specific spectra can be observed due to differences in raw materials and process. Genuine leather involves several tanning process, while imitation leather usually uses plastisol processing (Syabani et al., 2020). Some critical spectra of genuine and artificial leather are shown in Table 1.

Table 1. Infrared Characteristic Peaks of Genuine and Artificial Leather

Category	Wavelength (cm^{-1})	Type of vibration	References
Genuine leather	2800–3000	C−H stretching	(Pullawan, 2016)
	1650–1675	C=O stretching (amide I)	
	1540–1560	N−H bending and C−N stretching (amide II)	
	1230–1350	N−H bending, C−N stretching, C−C stretching (amide III)	
PVC-based artificial leather	2914–2950	CH_2 asymmetric stretching	(Ma et al., 2019)
	1430	CH_2 stretching	
	1254–1333	C−H deforming	
	960	CH_2 rocking	
	615–700	C−Cl stretching	

These spectral features provide valuable information about genuine leather's chemical composition and structural characteristics, allowing for its identification and differentiation from other materials. The FTIR analysis results are shown in Figure 4.

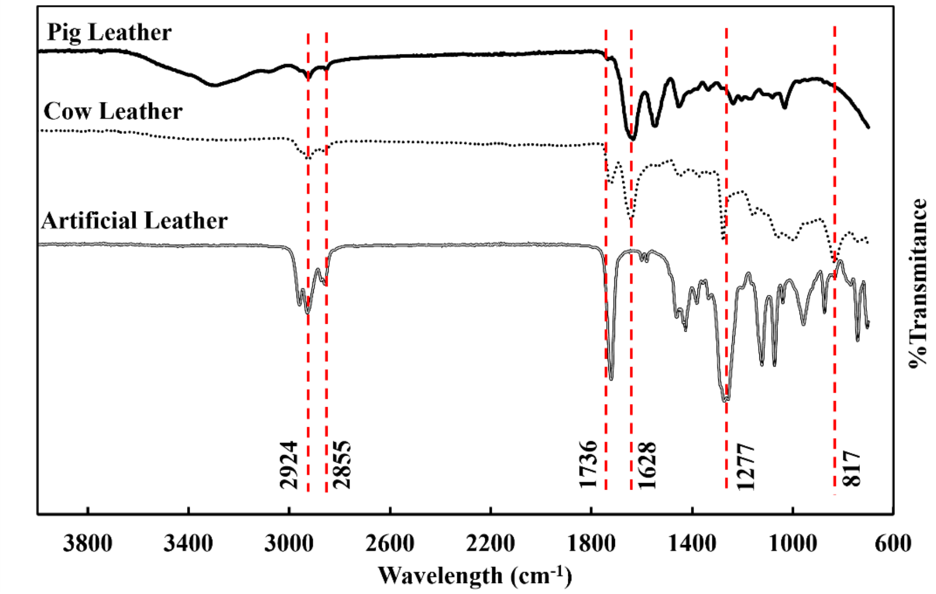


Figure 4. Leather FTIR spectra.

This qualitative analysis discovered that the spectra of pig and cow leathers were nearly identical. This similarity is attributed to the fact that leather primarily comprises collagen, a protein that forms the main structural component of skin (Hou et al., 2023). There are peptide bonds in the skin tissue of genuine leather that makes it relatively hard to distinguish between pig and cow leather. The amide I band appears in 1628 cm^{-1} for pigskin and 1642 cm^{-1} for cowhide. This spectrum is associated with the stretching vibration of the C=O bond in the amide groups present in proteins, such as collagen, a primary leather component (Sakmat et al., 2015). The amide II band appears in 1543 cm^{-1} and 1534 cm^{-1} for pigskin and cowhide respectively attributed to the combination of N–H bending and C–N stretching vibrations in the amide groups. The amide III bands occur in slightly different regions of 1234 cm^{-1} for pigskin and 1372 cm^{-1} for cowhide, representing a combination of N–H bending and C–N stretching vibrations and C–C stretching vibrations in the protein backbone. Lastly, the C–H stretching bands observed in the region of 2855–2924 cm^{-1} for pigskin and 2851–2919 cm^{-1} for cowhide correspond to the stretching vibrations of C–H bonds present in the aliphatic hydrocarbon chains of lipids and proteins in leather.

Although the spectra exhibit similarities, there are variations in the intensity of vibrations in certain regions. These distinctions can be employed effectively to differentiate between halal leather and pigskin leather. In the 1500–1800 cm^{-1} spectral range, commonly for peptide carbonyl group wavelength, both cowhide and pigskin display three similar peaks (Carsote et al., 2021). However, at a wavenumber of 1736 cm-1, the peak observed in pigskin leather appears faint and less sharp compared to the distinct peak observed in cowhide leather. Conversely, at a wavenumber of 1534 cm^{-1}, the peak obtained from cowhide leather appears faint and less pronounced compared to the sharp peak observed in pigskin leather spectra. Cowhide exhibits stronger vibrations at 1277 cm^{-1} and 817 cm^{-1} than pigskin.

As for artificial leather, the spectra obtained from PVC, which are non-leather materials, exhibited distinct variations with different peaks, clearly distinguishing them from natural cowhide or pigskin leathers. The FTIR spectra of PVC-based artificial leather typically exhibit characteristic peaks useful for identification. These peaks include stronger stretching vibrations of C–H in the range of 2859–2960 cm^{-1}, stretching vibrations of C=O around 1723 cm^{-1}, and stretching vibrations of C–Cl around 744 cm^{-1} (Pandey et al., 2016). The presence of unique C–Cl stretching spectra indicates the presence of PVC in the artificial leather material (Maia et al., 2017). Therefore, FTIR spectroscopy is an effective and efficient method for differentiating genuine leather and non-leather products during leather authentication (Pullawan, 2016).

3.3. Thermal Analysis

The thermal analysis used in this study is differential scanning calorimetry (DSC). In leather, the heat flow curve from DSC can be used to predict leather's denaturation, melting processes, and resistance to heat treatment (Cucos et al., 2014). Two primary temperature transitions are observed from 10°C to 445°C. The significant endothermic peaks (peak I) occurring between 10°C and 120°C, attributed to dehydration and thermal denaturation of collagen, exhibit overlapping characteristics. The highest peak corresponds to the denaturation temperature (Td), which signifies the transformation of the collagen triple helix in the leather into a random coil structure, whereas the onset was taken as the shrinkage temperature of the sample (Jeyapalina et al., 2007; Onem et al., 2017; Sebestyén et al., 2022). Moreover, DSC can also indicate the melting temperature (Tm) associated with the crystalline network strength (Zhang et al., 2021). The DSC analysis results for cowhide, pigskin, and artificial leather are presented in Figure 5.

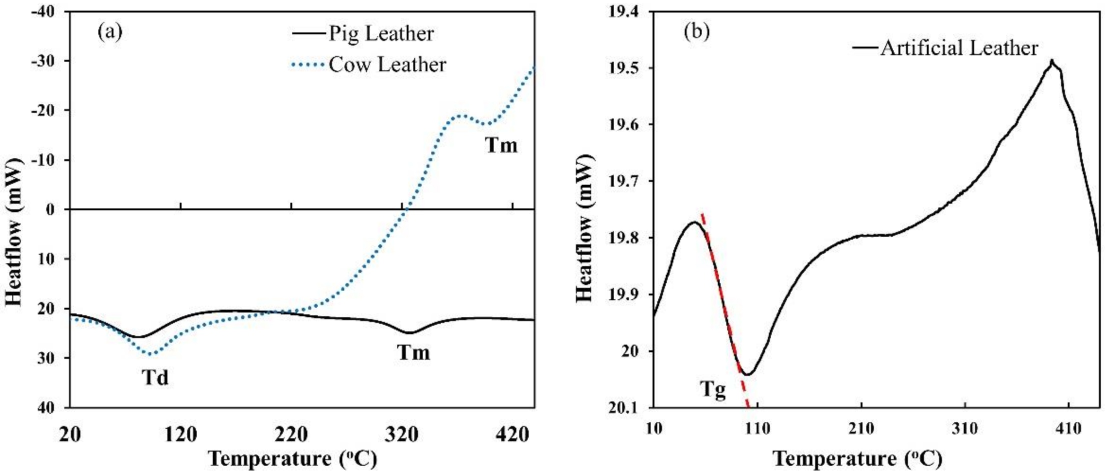


Figure 5. DSC curve of the genuine leather (a) and artificial leather (b).

In Figure 5a, two endothermic peaks are observed on the pig and cowhide leather DSC curve. The first peak indicates the leather's denaturation temperature (Td), and the second peak indicates collagen network decomposition (Tm) (Valeika, 2020). The weakening of the mechanical stability of the leather occurs as the fiber network of the grown tissue undergoes degradation (Meyer et al., 2021; Miao et al., 2021). Cowhide and pigskin leather have similar chemical compositions as they are both animal skin types. Despite this, we can still distinguish between different types of leather based on their thermal stability, as various tanning agents exhibit varying degrees of bonding strength with collagenous material (Onem et al., 2017). The cow leather exhibit higher Td and Tm value than the pig leather. However, the materials type is not the only factor that influences this value, but the tanning process also should be considered (Bañón et al., 2021; Fathima et al., 2010). The number of cross-linkages formed during tanning determines the denaturation temperature of the leather (Wibowo & Syabani, 2015; Witt et al., 2021). Hence, the thermal denaturation parameter serves as an indicator of leather's resistance to heat, so a higher Td and Tm value signifies improved thermal stability in leather (Carsote et al., 2021).

On the other hand, DSC analysis can easily distinguish between genuine and artificial leather. Artificial leather also gives two peaks on its curve, but the second peak is exothermic, as shown in Figure 5b. The first peak indicates the glass transition temperature (Tg) of PVC, which is the temperature at which PVC transitions from a glassy state to a rubbery state. This transition is often associated with changes in the polymer's mechanical and thermal properties (Jia et al., 2017; Tomaszewska et al., 2021). The second exothermic peak indicating the temperature at which thermal degradation or decomposition occurs, reveals the thermal stability of PVC.

Table 2. DSC Data of Leather Sample

Leather Sample	1st peak			2nd peak		
	Onset (°C)	Peak (°C)	Entalphy (J/g)	Onset (°C)	Peak (°C)	Entalphy (J/g)
Pigskin	42.67	80.01	282.19	294.36	324.36	130.29
Cowhide	46.22	89.76	241.82	372.50	401.16	224.70
Synthetic	51.67	95.84	11.65	366.62	391.90	-31.90

The denaturation temperature of cowhide leather is slightly higher than that of pigskin leather, with values of 89.76°C and 80.01°C, respectively. The enthalpy for pigskin leather is higher than that of cowhide leather, measuring 241.8236 J/g and 282.190 J/g, respectively. The denaturation temperature (Td) indicates the physiochemical changes that occur in the collagen structure of the leather, transitioning from a triple helix to a random coil. Lower denaturation temperatures suggest a weaker triple helix in the collagen molecule, indicating a less stable structure (Cucos et al., 2014). It should be noted that further research is required, considering the potential influence of the tanning process, as higher Td values could be attributed to increased hydrogen bonding and cross-linking resulting from collagen filling and the effects of tanning agents during treatment (Onem et al., 2017). Various binding mechanisms and bond strengths are exhibited by the tanning agents employed during leather production (Wibowo & Syabani, 2015). In contrast, PVC, as the primary material used in artificial leather, does not undergo cross-linking. Consequently, the first peak in the DSC curve of PVC exhibits a significantly lower enthalpy of 11.65 J/g, corresponding to its glass transition temperature. Using plasticizers to make artificial leather might also contribute to its lower enthalpy (Syabani et al., 2020). These plasticizers reduce the intermolecular forces between polymer chains, allowing them to move more freely (Lim & Hoag, 2013). As a result, PVC's glass transition temperature (Tg) is lowered, and the polymer transitions from a rigid glassy to a more flexible rubbery state.

The second endothermic peak observed in Table 2 corresponds to the melting process of the leather. The melting temperature (Tm) of the crystalline collagen zone indicates the crystalline phase's thermal stability, which relates to the amorphous-crystalline structure of collagen (Vyskočilová et al., 2022; Zhang et al., 2021). Cowhide leather exhibits a higher melting temperature than pigskin leather, with values of 401.16°C and 324.36°C, respectively. The lower melting temperature in pigskin leather might be associated with lower cross-linking in collagen-based materials and their degradation (Onem et al., 2017). Although the second peak temperature of artificial leather may have a similar value to that of genuine leather, the enthalpy was negatives that representing exothermic processes. This decomposition process of PVC includes eliminating hydrogen chloride and releasing small molecules such as CO and CO_2 (Ma et al., 2019). By comparing the DSC curves of an unknown sample with reference curves of known animal leather and PVC artificial leather, it becomes possible to differentiate between the halal materials based on their distinct thermal behaviors and transitions.

4. Conclusion

In summary, SEM, FTIR, and DSC techniques can be utilized to determine the origin of the raw materials used in leather products. Surface analysis through visual examination aids rapidly in distinguishing between different types of leather based on grain patterns, pores, and the presence of the three-hole pattern in pig leather. In contrast, artificial leather can be identified by its plain or uniform formation of breathing holes and the use of a fabric layer. However, contemporary leather finishing techniques can diminish the accuracy of surface morphology. Therefore, Fourier Transform Infrared Spectroscopy (FTIR) offers rapid and reliable analytical techniques that can be used to identify leather types. The spectra of cowhide and pigskin leathers show similarities, but differences in vibration intensity allow for practical distinction. Artificial leather, such as PVC-based materials, exhibits distinct spectra, making FTIR spectroscopy valuable for distinguishing between halal and non-halal leather products. Cowhide displays intense vibrations at wavenumbers 1736 cm^{-1}, 1277 cm^{-1}, and 817 cm^{-1}, whereas pigskin exhibits stronger vibrations at 1534 cm^{-1}. On the other hand, artificial leather made from PVC shows notable stretching at 1723 cm^{-1} and 744 cm^{-1} wavenumbers. Differential scanning calorimetry (DSC) analysis can distinguish between genuine and artificial leather based on distinct peaks and their thermal behaviors. However, differentiating between cowhide and pigskin leather using DSC may not be straightforward due to their similar thermal properties. The denaturation temperature of cowhide leather is slightly higher than that of pigskin leather, with values of 89.76°C and 80.01°C, respectively. The enthalpy for pigskin leather is higher than that of cowhide leather, measuring 241.8236 J/g and 282.190 J/g, respectively. Artificial leather, such as PVC-based materials, exhibits unique thermal characteristics, including a glass transition temperature (Tg) and exothermic peak. For future research, it is suggested to apply this methodology to analyze other types of leather, including different types of pigskins, reptiles, fishes, sheep, and goats, to gain a more comprehensive understanding.

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