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# Hydrophobic Coating of Vegetable-Tanned Leather with Dodecylamine

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## Article

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## ABSTRACT

*Vegetable-tanned leather exhibits a hydrophilic character due to the abundance of hydroxyl groups (-OH). In this study, the surface of vegetable-tanned leather was modified to become hydrophobic. The leather was coated using dodecylamine (DDA) through the padding method in the finishing process. The concentration of DDA varied from 1%, 2%, 3%, and 4% to identify the optimum concentration. The coating process was assisted by an activator, either acid or base, to improve the adhesion of DDA to leather. Uncoated vegetable-tanned leather was used as a control. The success of coating was identified using FT-IR and SEM-EDX analysis. The hydrophobicity of leather was conducted through a water resistance test. The success of coating is shown by the increase in their weight and thickness. Absorption bands of alkyl and amino groups of DDA are observed in FT-IR spectra around 1450 cm<sup>-1</sup> and 3400 cm<sup>-1</sup>, respectively. SEM micrographs show that coated leathers have flatter surfaces as a consequence of DDA attachment on the leather surface. The water resistance test proves that the use of DDA can improve the leather hydrophobicity and enhance the water absorption time by up to 4 times. The optimum water absorption time is obtained when using 2% DDA.*

## KEYWORDS

*vegetable-tanned leather, dodecylamine, hydrophobic coating, water resistance*

## INTRODUCTION

Currently, the leather tanning process still widely uses chrome(III) as a tanning agent. However, chrome(III) is known to be easily oxidized to chrome(VI), so its use should be minimized. One alternative tanning agent is a vegetable tanning agent. Vegetable tanning agents contain polyphenols so they have lots of hydroxyl (-OH) groups. This causes vegetable-tanned leather to have hydrophilic properties [1]. The hydrophilicity of leather increases the ability to absorb water. Dirt carried by water can stick to the surface of leather, making it easily dirty and becoming a good medium for bacterial growth. Therefore, it is necessary to alter the leather surface to become hydrophobic.

The application of leather in various goods, such as footwear and garments, requires the leather to have water-resistant properties. Water-resistant properties can be created by imitating the

superhydrophobic character of taro leaf structure [2]. Taro leaf is composed of a hierarchical structure with micropapillae formed by the self-assembly of long-chain hydrocarbon waxes that make the surface hydrophobic [3]. Several ways to improve the hydrophobicity of leather can be conducted using a water-repellent material in the retanning, fatliquoring, or finishing process. Its use in the retanning or fatliquoring process allows the material to penetrate through the cross-section and collagen fibres to be coated perfectly. Meanwhile, its use in the finishing process produces a hydrophobic layer on the leather surface [4].

Leather coating in the finishing process is considered easier and more efficient because it requires less time and supporting chemicals. Many alternative coating methods have been developed to obtain hydrophobic surfaces, including plasma polymerization [4-7] and spraying methods [8]. One finishing method that is often applied is the padding method. In this method, the chemical used can be transferred optimally onto the leather [9].

Several studies have reported surface modifications with silica and fluoro compounds, both on natural and synthetic leather [3,8,10]. However, fluorinated compounds have negative effects on the environment, including contamination, bioaccumulation, and high toxicity [11]. Organosilane has been developed to produce hydrophobic leather with a water contact angle of 135-140° [4,10,12]. Its hydrophobic character is ascribed to the long-chain hydrocarbon of organosilane. Likewise, dodecylamine (DDA) has been reported to provide hydrophobic, water-repellent, and self-cleaning characteristics [2,13,14]. DDA contains an amino group that interacts with water molecules strongly and a long-chain hydrocarbon that provides hydrophobic properties [14]. This hydrophobic character is the reason for using DDA in leather coating material in this study. Modification of vegetable-tanned leather with DDA is assisted by an activator to improve adhesion. Two types of activators, which are acid and base, are selected to investigate the most optimal for use in the leather coating process.

## EXPERIMENTAL

### Materials and Methods

#### *Materials*

Materials used were mimosa-tanned goat leathers (produced by Politeknik ATK Yogyakarta), isopropyl alcohol 95% (technical grade), citric acid (PT Golden Sinar Sakti), ammonia (technical grade), and dodecylamine (DDA, Merck).

#### *Methods*

The coating process was done for leather finishing by padding method. Tanned leathers were cut into pieces of 15 x 15 cm and then wiped with 5 mL of isopropyl alcohol for clarity. After drying at room

temperature, every piece of leather was wiped using 5 mL of an activator (acid/base) and then dried at room temperature. About 5 mL of DDA solution in various concentrations was wiped onto it and then dried (Table 1). The coated leathers were characterized using FT-IR analysis (Perkin Elmer 96681) [15-18], SEM-EDX analysis (JEOL JSM-6510LA) [1,19,20], and water resistance test.

To identify the functional groups of leather, the sample was cut to 0.5 x 0.5 cm. This piece was put in the sample holder of an FT-IR spectrophotometer and the absorbance was measured at a wave number range of 400-4000  $\text{cm}^{-1}$ . The morphology and elemental content of leather were determined using SEM-EDX with 20 kV power and 100  $\mu\text{m}$  magnification. A drop test method was carried out to identify the water resistance of leather. A 5  $\mu\text{L}$  of water droplet was dripped on the surface of the leather at a distance of 3 cm. The water absorption time was determined.

Table 1. Variations in the use of chemicals for coating process

Sample	Activator	Concentration of DDA (%wt)
Control	-	-
A1	Acid (Citric acid 2.5%)	1
A2	Acid (Citric acid 2.5%)	2
A3	Acid (Citric acid 2.5%)	3
A4	Acid (Citric acid 2.5%)	4
B1	Base (Ammonia 2.5%)	1
B2	Base (Ammonia 2.5%)	2
B3	Base (Ammonia 2.5%)	3
B4	Base (Ammonia 2.5%)	4

## RESULTS AND DISCUSSION

### *Coating Process*

Adhesion plays an important role in the finishing process attributing to the interaction of leather surface and finishing chemical. Generally, adhesion in the finishing process occurs with a combination of mechanisms. Adhesion mechanisms that may be found are mechanical adhesion, chemical bonding, and diffusion adhesion [21].

Hydrogen ions ( $\text{H}^+$ ) from acid activator make the surface of leather positively charged. Likewise, DDA has a positive charge attributed to an amino group [22]. This similarity charge causes good penetration and leads to slow adhesion on the leather surface [23-25]. After contacting and penetrating the leather, DDA binds to the leather through hydrogen bonding. In this case, the possible adhesion mechanisms are mechanical adhesion and chemical bonding. The hypothetical hydrogen bonding that forms is presented in Figure 1. Hydrogen bonding is included in chemical bonding even though it is

considered a weak interaction. The large number of these bondings on the leather surface still has a significant effect [21].

Ammonia as a base activator produces hydroxide ions ( $\text{OH}^-$ ) and provides a negative charge to leather. Consequently, the leather charge which was initially positive resulting from its pH being below the isoelectric point (IEP), changes to become negative. The opposite charges between the leather and DDA prevent DDA from penetrating the leather, resulting in the formation of hydrogen bonding on the leather surface directly [23]. Thus, the adhesion mechanism when using a base as an activator is chemical bonding. Therefore, it is concluded that acid and base can be used as activators in the leather coating process with DDA despite different levels of penetration depth.

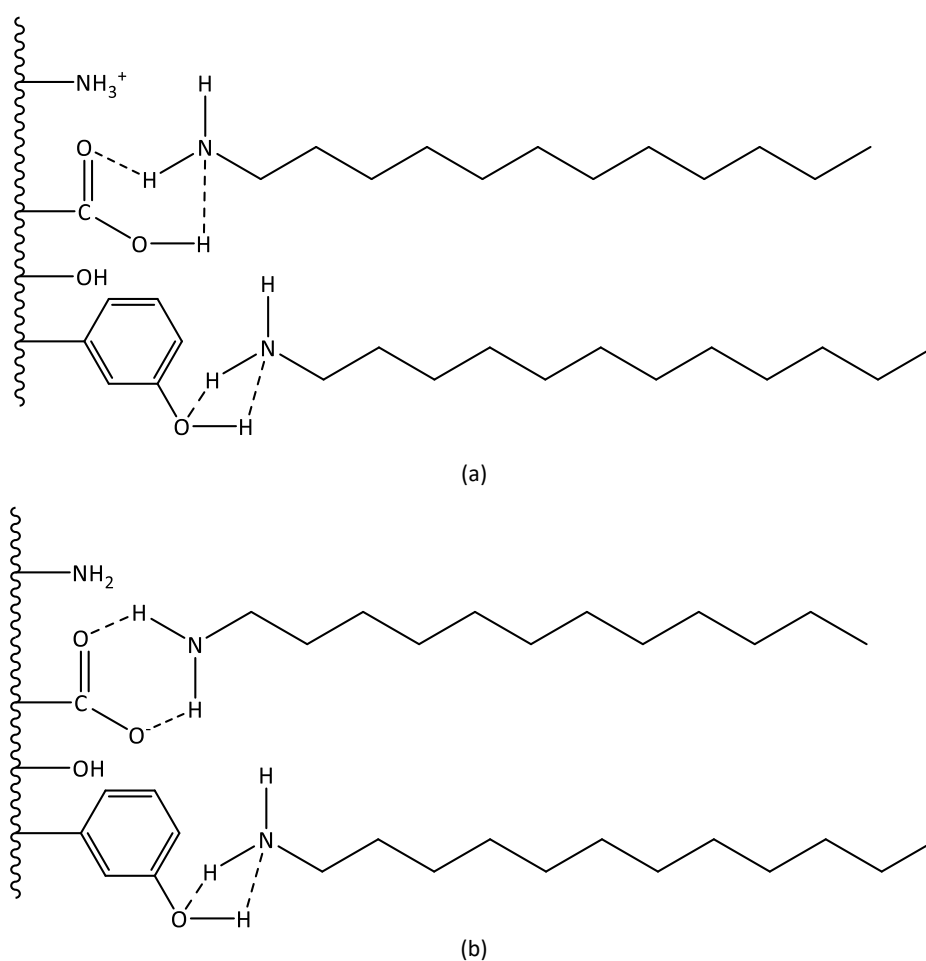


Figure 1. Hypothetical structure of coated vegetable-tanned leather: (a) using an acid activator and (b) using a base activator

### Physical Properties

Modification of leather using DDA results in an increase in its weight and thickness. Table 2 shows that the weight of every treatment has increased but not significantly. Likewise, there is an increase in its

thickness but it is not significant. This phenomenon occurs because of the small amount of DDA used. However, this implies the success of leather coating.

Table 2. Weight and thickness of leathers modified with DDA

Sample	Weight (g)			Thickness ( $\mu\text{m}$ )		
	Before	After	Difference	Before	After	Difference
A1	13.81	13.84	0.02	$720.2 \pm 58.7$	$728.2 \pm 54.8$	8.0
A2	15.19	15.21	0.02	$789.2 \pm 74.8$	$796.2 \pm 77.3$	7.0
A3	16.42	16.56	0.14	$946.4 \pm 74.9$	$955.8 \pm 65.9$	9.4
A4	14.72	14.78	0.06	$840.3 \pm 110.6$	$852.7 \pm 66.6$	12.3
B1	13.23	13.24	0.01	$720.0 \pm 27.2$	$721.7 \pm 29.3$	1.7
B2	17.20	17.37	0.18	$1029.8 \pm 82.1$	$1037.6 \pm 81.3$	7.8
B3	15.68	15.73	0.05	$917.0 \pm 43.9$	$928.6 \pm 47.7$	11.6
B4	14.46	14.57	0.10	$807.6 \pm 64.8$	$822.6 \pm 44.1$	15.0 <sup>s</sup>

Note: s: there is a significant difference between the two groups ( $P < 0.05$ )

### Functional Groups Identification

Infrared spectra of control and leather modified with DDA can be seen in Figure 2. Characteristic absorptions of vegetable-tanned leather are observed from the presence of amide, methylene, and hydroxyl groups. Amide I appears at a wavenumber range of  $1642\text{--}1651\text{ cm}^{-1}$  as a carbonyl group ( $\text{C=O}$ ) stretching vibration [1,17,26]. The vibration band at  $1539\text{--}1558\text{ cm}^{-1}$  is a result of overlapping between C-N of amide II stretching and N-H bending [1,27,28]. Amide III is shown at a range of  $1234\text{--}1236\text{ cm}^{-1}$  from carboxylate group stretching [17,28,29]. The methylene group is detected at  $1455\text{--}1458\text{ cm}^{-1}$  from its bending vibration [18]. Furthermore, absorption of C-H asymmetric stretching vibration appears at  $2926\text{--}2927\text{ cm}^{-1}$  [30]. Vibration broadband at  $3339\text{--}3406\text{ cm}^{-1}$  is identified as O-H stretching [31]. Stretching of C-O-C from vegetable tannin results in a band at  $1033\text{--}1090\text{ cm}^{-1}$  [17,32].

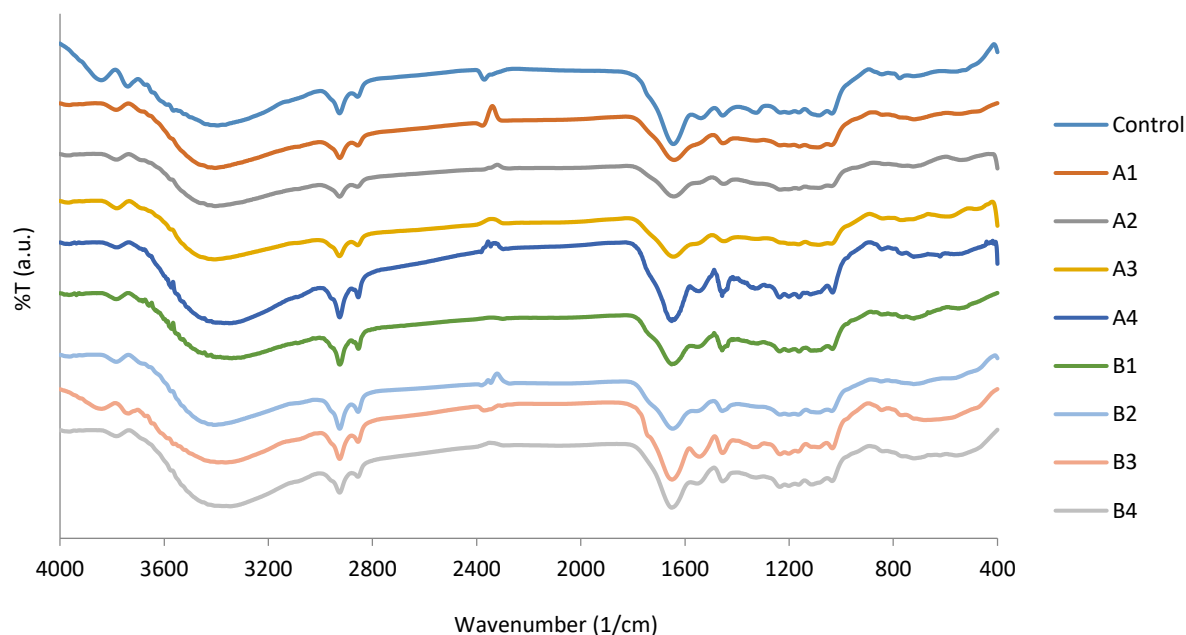
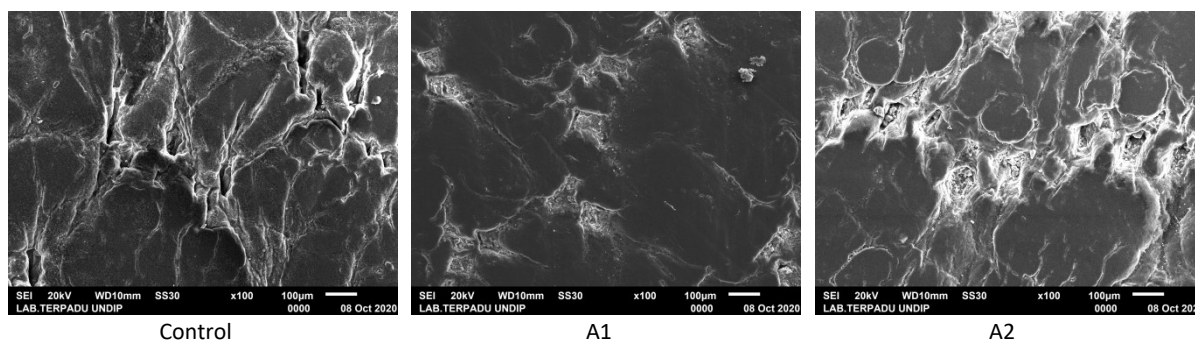


Figure 2. FT-IR spectra of leathers: control, A1, A2, A3, A4, B1, B2, B3, and B4

The presence of DDA on the leather results in slightly different intensities of the absorption band. This is due to the same functional group content of DDA as the leather which exhibits methyl, methylene, and amino groups. When compared with the control absorption band, the alkyl groups of DDA give a higher intensity of absorption band at  $1455\text{--}1458\text{ cm}^{-1}$ . This proves the success of DDA attachment to the leather. Characteristic absorption of the amino group is commonly observed at  $1628\text{ cm}^{-1}$  and  $3426\text{ cm}^{-1}$  as bending and stretching of -NH bond, respectively [33]. In this research, that absorption band overlaps with -OH of leather vibration. However, the absorption bands around  $3400\text{ cm}^{-1}$  are observed to become wider as the amount of DDA used increases. Moreover, Figure 2 shows that there is no difference in the absorption band for the use of acid or base in the modification process. This indicates that both can be used for the leather modification process with DDA.

### Morphology and Elemental Analysis



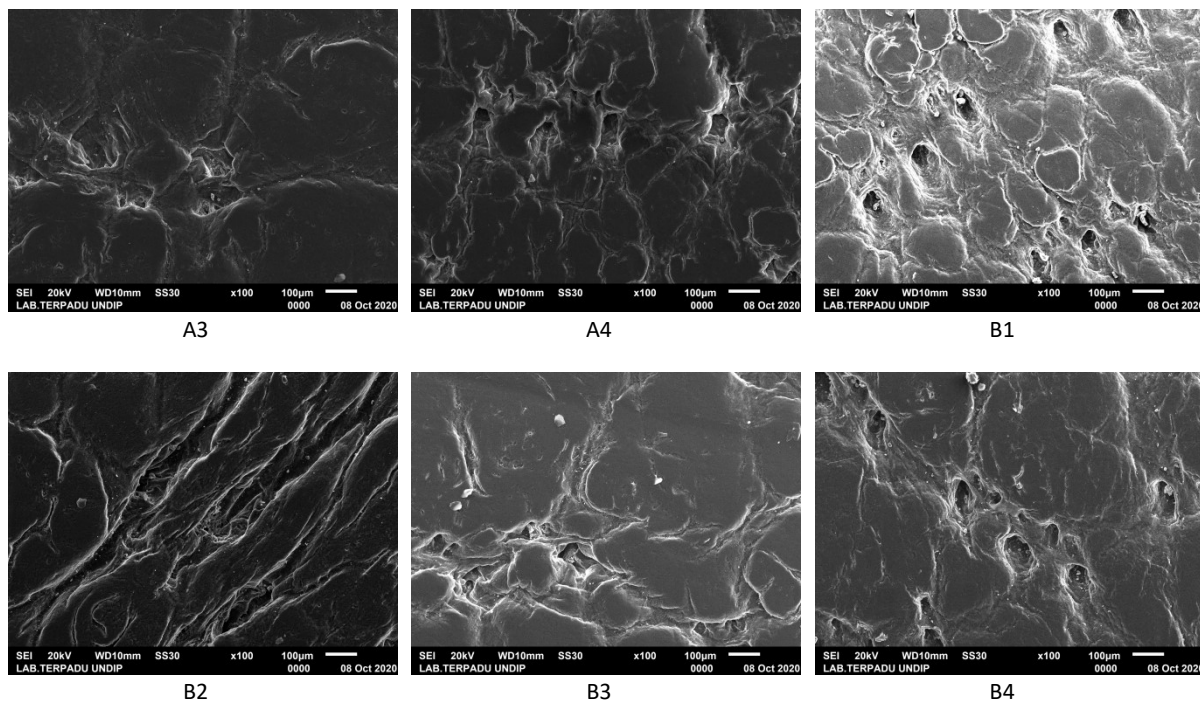


Figure 3. SEM images of leathers: control, A1, A2, A3, A4, B1, B2, B3, and B4

SEM micrographs indicate that the control has more cavities than modified leathers (Figure 3). Meanwhile, the modified leather exhibits a flatter surface due to the cavity closure by DDA. The micrographs show that base-activated leathers have more porous surfaces than acid-activated leathers. This is caused by the alkali swelling that opens up the fibres' structure as a consequence of the opposite charges between the leather and DDA when using a base activator [21].

The elemental content of leather is written in Table 3. It is known that the highest content of leather, both control and modified leather, is carbon (C). The presence of DDA hydrocarbon groups generally causes an increase in the percentage of C in modified leather compared to the control. However, it is seen that there is a decrease in C content for A1 and A2, which is aligned with the increase of O and Si content. This phenomenon allegedly occurs due to the presence of impurities during the process.

Table 3. Elemental content of leather

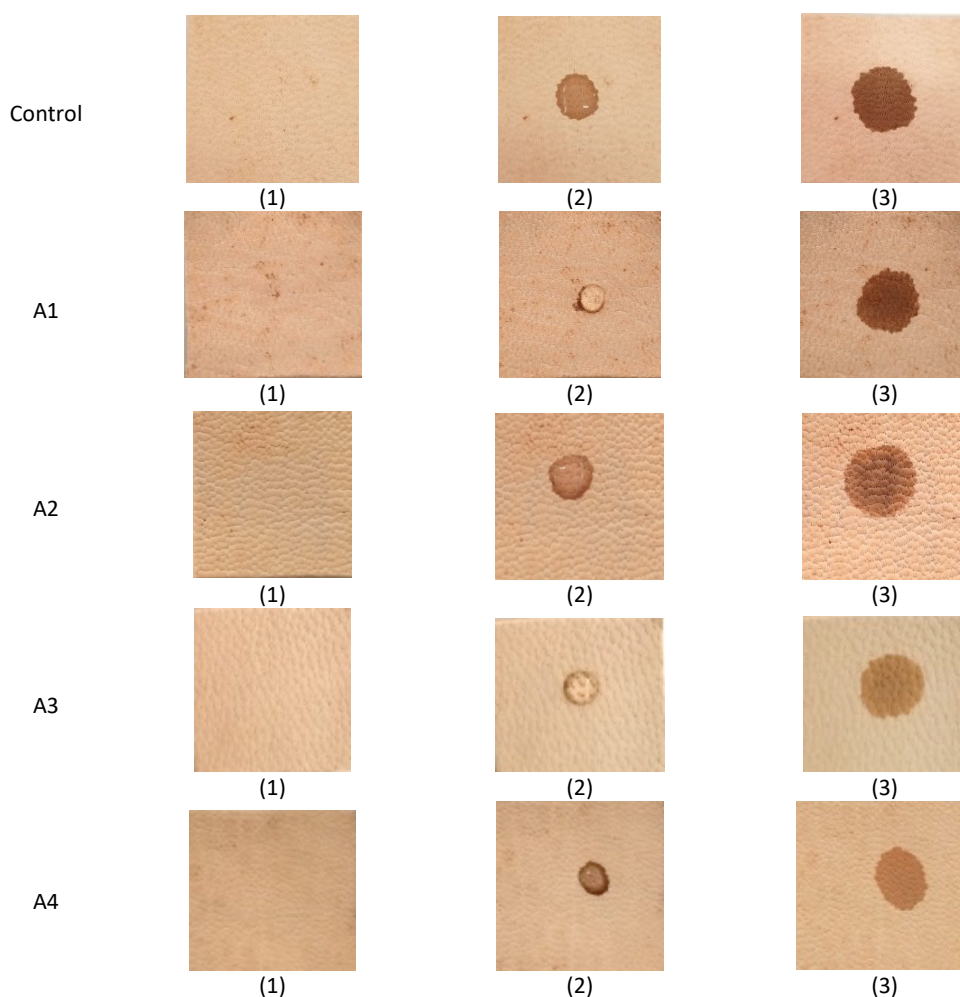
Sample	Chemical composition (%wt)					
	C	O	Na	Si	S	Ca
Control	63.23	33.23	2.16	0.08	1.00	0.15
A1	61.29	34.42	2.21	0.75	1.02	0.15
A2	62.10	33.78	1.55	1.16	0.99	0.32
A3	67.92	29.82	0.65	0.34	0.84	0.36
A4	65.56	32.49	0.98	-	0.89	0.08
B1	64.69	32.40	1.01	0.29	0.99	0.19
B2	70.01	26.91	-	0.15	1.05	0.84



Sample	Chemical composition (%wt)					
	C	O	Na	Si	S	Ca
B3	68.55	29.47	0.77	0.10	0.95	0.16
B4	64.52	32.38	0.89	0.12	0.97	0.27

### Water Resistance Properties

A drop test was carried out to determine the effect of DDA on the water resistance of leather. This method is considered easier and faster than the contact angle measurement method [34]. The appearance of leather can be seen in Figure 4. The use of vegetable tanning agents leads to the hydrophilicity of leather so that the contact angle between the water droplet and the leather surface is less than  $90^\circ$ . The hydrophilicity of leather remains visible with perfect water absorption into the leather (Figure 4). However, the results of water absorption time show that the use of DDA can increase the water absorption time up to 4 times (Table 4). This is under the hypothesis that the presence of a hydrocarbon chain in DDA can increase the hydrophobicity of leather. This causes water to be retained on the surface of the leather. Therefore, it is concluded that DDA is effective as a water-repellent agent for leather.



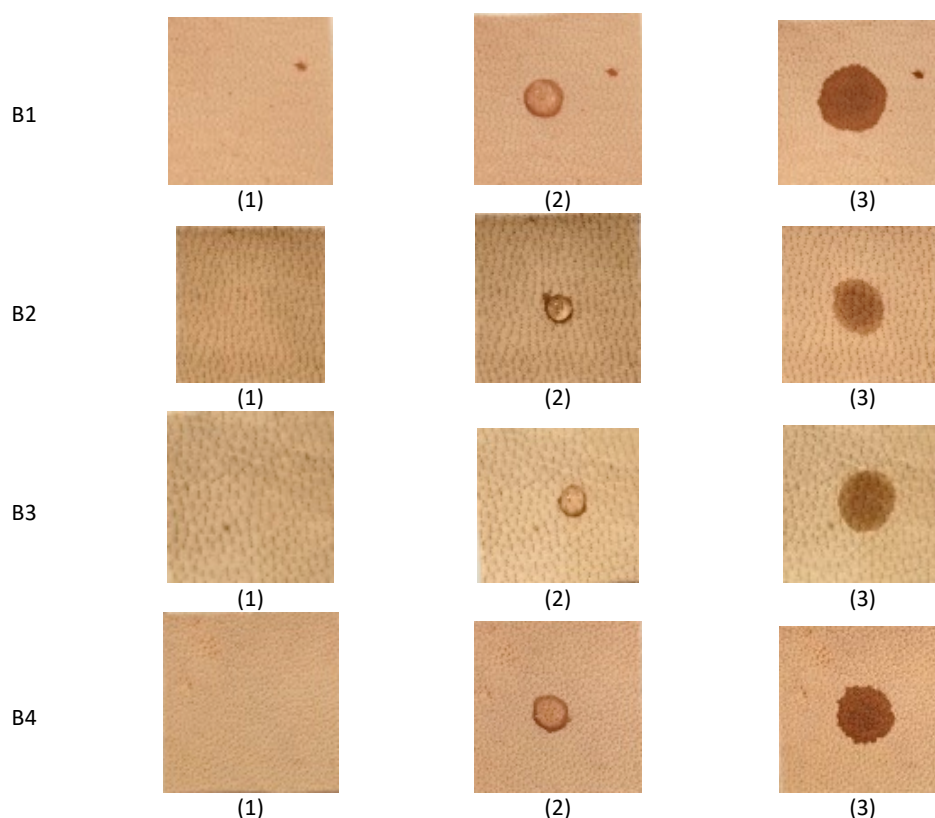


Figure 4. Visual of leathers: control, A1, A2, A3, A4, B1, B2, B3, and B4; (1) before dripping water, (2) after dripping water, and (3) after absorbing water

Table 4. Time for water absorption

Sample	Absorption time (s)
Control	$23.98 \pm 1.82^a$
A1	$75.77 \pm 14.21^c$
A2	$106.24 \pm 10.81^d$
A3	$70.04 \pm 7.46^{b,c}$
A4	$51.31 \pm 6.93^b$
B1	$70.40 \pm 7.28^{b,c}$
B2	$110.86 \pm 17.99^d$
B3	$110.59 \pm 22.09^d$
B4	$99.86 \pm 18.42^d$

Note: a, b, c, and d show real differences ( $P < 0.05$ )

Table 4. informs that the time for water absorption of leather increases with the use of DDA. This proves that the hydrocarbon chain of DDA is successfully attached to the leather and improves its hydrophobicity [35]. However, absorption time decreases after using 2% DDA for both acid or base activators. Excess amount of DDA over the number of active groups on leather leads to the inability of DDA to reattach to the leather [23].

## CONCLUSION

Coating of leather using DDA succeeded in increasing leather hydrophobicity which is proven by an increase in the absorption time of water on leather surface. The success of coating is shown by the increase in their weight and thickness. FT-IR and SEM-EDX analysis results also confirm that DDA was successfully attached to vegetable-tanned leather. A water resistance test proves that the use of DDA can improve the leather hydrophobicity and enhance the water absorption time by up to 4 times. The optimum water absorption time is obtained when using 2% DDA. However, it is still necessary to study the release ability of DDA from leather.

## Author Contributions

Conceptualization – Rosiati NM; methodology – Rosiati NM; formal analysis – Udkhiyati M and Silvianti F; investigation – Rosiati NM and Udkhiyati M; resources – Rosiati NM and Silvianti F; writing-original draft preparation – Rosiati NM and Udkhiyati M; writing-review and editing – Rosiati NM, Udkhiyati M and Silvianti F; visualization – Rosiati NM and Udkhiyati M; supervision – Rosiati NM. All authors have read and agreed to the published version of the manuscript.

## Conflicts of Interest

The authors declare no conflict of interest.

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