A PRELIMINARY STUDY ON THE APPLICATION OF EUCALYPTUS OIL FOR ANTIBACTERIAL PROPERTIES ON COTTON/POLYESTER FABRICS

STUDI AWAL PENGGUNAAN MINYAK KAYU PUTIH UNTUK SIFAT ANTIBAKTERI PADA KAIN KAPAS/POLIESTER

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ABSTRACT

Essential oils as the antibacterial substance in textiles are commonly demanded since they are easily found and have additional properties as relaxation therapy. This study aims to receive an antibacterial cloth using eucalyptus oil as a finishing substance gained from the leaves of *Melaleuca leucadendra L*. from a local industry (Perhutani Gundih, Central Java). The four types of cotton/polyester fabrics were prepared for finishing treatment with the bathing direct application method. They were 100% cotton, 100% polyester, 80%/20% polyester/cotton, and 65%/35% polyester/cotton. FTIR findings proved the existence of the eucalyptus main compound (α -pinene) in those four fabrics. Moreover, the antibacterial tests using *S. aureus* cultivation showed that only 100% polyester-treated fabric did not obtain antibacterial properties because the diameter of bacterial inhibition was zero millimeters. The other three fabrics potentially have antibacterial properties even if they did not gain a high diameter inhibitor zone.

Keywords: antibacterial; eucalyptus oil; cotton fabric; finish

ABSTRAK

Minyak atsiri sebagai zat antibakteri pada tekstil banyak diminati karena mudah ditemukan dan memiliki khasiat tambahan sebagai terapi relaksasi. Tujuan dari penelitian ini adalah menghasilkan kain dengan sifat antibakteri menggunakan minyak kayu putih sebagai zat penyempurnaan yang diperoleh dari daun Melaleuca leucadendra L. dari industri lokal (Perhutani Gundih, Jawa Tengah). Keempat jenis kain kapas/poliester tersebut disiapkan untuk finishing dengan metode aplikasi rendaman langsung. Jenis kain sampel adalah kapas 100%, poliester 100%, polyester/kapas 80%/20%, dan poliester/kapas 65%/35%. Temuan FTIR membuktikan adanya senyawa utama kayu putih (a-pinene) pada keempat kain tersebut. Selain itu, uji antibakteri menggunakan kultivasi S. aureus menunjukkan bahwa hanya kain poliester 100% yang tidak memperoleh sifat antibakteri karena diameter daya hambat bakteri menunjukkan nol milimeter. Tiga kain lainnya berpotensi memiliki sifat antibakteri meskipun tidak memperoleh zona hambat berdiameter tinggi.

Kata kunci : antibakteri, minyak kayu putih, kain kapas, finish

INTRODUCTION

The use of natural materials as antimicrobial agents in textiles is becoming increasingly in demand in the industry because, with natural materials, the production will be relatively safe, non-toxic, and will not impact the environment negatively.^{1–4} Generally, these natural ingredients are made from plant extracts such as aloe vera, tea tree, eucalyptus, neem, grape seeds, and tulsi leaves, as well as essential oils with important substances in them such as phenols, flavonoids, and tannins. ^{5–7} According to Borris (1996), there are more than 5

million plant species on earth, with 1% of them being plants with medicinal benefits and properties.⁸ Other plants that are used, apart from being antimicrobial, have also been used in several previous studies as dyes as well as protection against ultra-violet (UV) rays, namely extracts from saffron, green tea, onion peel, and eucalyptus.^{9–11} Several studies had different perceptions of the name *Melaleuca leucadendra L*. The studies of Kartiko *et al.* (2021) and Helfiansyah *et al.* (2015) mentioned the oil of *Melaleuca leucadendra L*. as cajuput oil.^{12,13} In contrast, four other research publications referred to *Melaleuca leucadendra L.* oil as eucalyptus oil and translated it into Bahasa Indonesia as *minyak kayu putih.* ^{14–17} This study, like the other four, utilized the phrase "eucalyptus oil", which also recognized by the local industry (Perhutani Gundih).

Essential oils in Indonesia are mostly used as therapy. Still, in textile research, this essential oil has the potential for a finishing process with antimicrobial benefits because there are still few published studies on this matter.¹⁸ In fact, this herbal product has advantages such as lowering the negative impact of chemical reactions compared to synthetic antimicrobial substances, and lowering production costs so that it can be explored more as an alternative to environmentally friendly antimicrobial substances.^{19,20}

Several previous studies have used eucalyptus oil as an antimicrobial in textile materials. ^{11,21–24} Research by Mongkholrattanasit et al. (2011) used eucalyptus leaf extract as a dye and ultraviolet (UV) ray protector with a brownish yellow color on silk fabrics.²⁴ They stated that this eucalyptus leaf extract produced more than 10% tannins (gallic acid and ellagic acid), flavonoids (quercetin and rutin) and, polyphenols. The bark produces color from the quercetin component. Ali et al. (2007) used bark extract of eucalyptus (bark) as a dye with sodium sulfate as a mordant to produce medium to good color resistance.²¹ The mordant. which is usually in the form of salt, is used to fix the fiber and dye so that a bond is formed between the two.^{25,26}

In line with the research of Mongkholrattanasit et al. (2011), groups of researchers like Ben Fadhel et al. (2012) and Silva et al. (2018) used eucalyptus leaf extract, but they used it on wool and cotton fabrics. As a result, the antimicrobial properties in this refinement reacted significantly. The UV protection properties were good, but their durability decreased with the number of washings. In addition to the finished fabric, eucalyptus oil is also applied to the manufacture of fibers, as was done in the study of Khajavi et al. (2014) for alginate fibers in wound dressing applications. They put eucalyptus oil in the fiber spinning process and produced good antibacterial properties on the fiber. 23

Using natural substances, both from plant extracts and essential oils, requires strong bonds such as covalent so that these substances will not easily disappear immediately when impregnated in textiles.²⁷ So, it is necessary to have a cross-linking agent or microencapsulation to increase the durability and control the release of the extraction of these substances. Microencapsulation is unique with tiny particles that coat and provide a blanket layer to protect the material on a micro-scale which can later work or release its function under controlled conditions.^{28–31} Research that uses essential oils with the microencapsulation method is Thilagavathi *et al.* (2007). They used neem oil (neem) as the core material and gum arabic as the capsule wall and applied it to cotton fabrics using the pad-dry-cure method.³² As a result, the microbial activity decreased, and SEM observations showed the microencapsulation adhered well to the fiber. In addition, washing up to more than 15 times still has a fairly good antimicrobial activity.

From the literature review above, the studies that had been done previously had several shortcomings that this research will contribute to: 1. Previous studies had used eucalyptus plants, but had not specifically used refined eucalyptus oil; 2. Previous studies had not included antimicrobial results on cotton fabrics with no refinement compared with only soaking with and without crosslink agents. With the shortcomings of prior research, this study aims to contribute by conducting: 1. Applying a locally produced eucalyptus oil (Perhutani Gundih, Central Java) as an antibacterial substance to the textiles; 2. Analyzing the effect of eucalyptus oil as an antimicrobial substance on different types of fabric made from natural or synthetic fibers or blended of both. The reason for choosing cotton and polyester for this study is that both fibers are the most common fibers used in domestic textiles such as tshirts, pants, and even cloth masks. Eucalyptus oil applied as an antibacterial substance is to reduce the use of synthetic substances in textile industries, which is also stated by previous studies. ^{23,24}

MATERIALS AND METHODS Materials

Plain weave cotton/polyester fabrics were used. They were cotton 100% from Primissima, 80/20 polyester/cotton with the trade name of Toyobo®, and 65/35 polyester/cotton from Nikita®. Table 1 shows the specifications of those fabrics. The reason for choosing those fabrics is that cotton and polyester are the most consumed fibers among natural and synthetic fibers. The oil of eucalyptus leaves (*Melaleuca leucadendra* L.) was used for the antimicrobial finish. The oil was purchased from Perhutani Gundih, Central Java, Indonesia.

 Table 1. Specifications of the samples

Sample ID	Composition	Density (g/m ²)	Warp/Weft (tpi)
СОТ	Cotton 100%	127,5	78/68
PE	Polyester 100%	63,9	90/75
TYB	Polyester	141,4	105/80
NKT	80%/Cotton 20% Polyester 65%/Cotton 35%	117,8	85/70

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Methods

The finishing treatment was obtained using eucalyptus oil as the core material and gum acacia as the wall material. This method was also adopted from the study by Thilagavathi et al. (2007).³² Gum acacia was mixed with 100 mL of hot water to allow it to swell for 30 minutes. Gum acacia had an excellent capability to be the wall of encapsulation due to its high solubility in water, low solution viscosity, good surface activity, and emulsification capability.³³ 50 mL of hot water was added to the mixture and stirred for 15 minutes at 40-50°C. Then 10 mL of eucalyptus oil was added to the mixture and stirred at 300-500 rpm for 15 minutes, followed by adding 20% of sodium sulphate and stirring for 10 minutes. Because of its role as a dissolving agent, sodium sulphate is commonly used in encapsulation production. The next step was adding 5 mL of formaldehyde with a 15% concentration while decreasing the stirrer speed. This solution was poured into the tray, the fabric was immersed and squeezed into it, then dried at 85°C in the oven. The eucalyptus oil was characterized by a Fouriertransform infrared spectroscopy (FTIR) test. The treated fabric was also examined by FTIR to analyze the presence of eucalyptus oil in the fabric. The treated fabrics were then observed with antibacterial tests according to SNI ISO 20743:2011. A SEM analysis was conducted to detect the spread of microcapsules on the fabric surface. The diameter was approximately measured using the software linked to the SEM. The measurement was conducted in at least five spots for each type of fabric sample.

RESULTS AND DISCUSSION FTIR Mapping Analysis

According to Knight (2009) and Alexander (2015), eucalyptus oil contained 60% 1,8-cineol and 20% α -terpineol and β -pinene.^{34,35} Therefore, the fabric samples were observed for the existence of 1,8-cineol based on its wavelength and absorbance. It was known from the research of Nyaga and Kamweru that the wavelength and absorbance of 1.8-cineol were 1377 cm⁻¹ and 0.0323. respectively.³⁶ On the other hand, the published work of the Cobletz Society (2018) figured the peaks of 1,8-cineol in the form of liquid and solvent.³⁷ They found one of the 1,8-cineol peaks was at the wavelength of 1015 cm⁻¹ with an absorbance of 0,285 and 1014-1016 cm⁻¹ with an absorbance of 0,240-0,243. Figure 1 shows the appearance of the 1,8-cineol peak on each fabric.



Figure 1. The FTIR mapping on four different types of fabrics with anti-microbial finishes using eucalyptus oil with a direct bathing application method: (a) 100% cotton, (b) 100% polyester, (c) 80%/20% polyester/cotton, and (d) 65%/35% polyester/cotton

The arrows in Figure 1 point out the peak of 1,8-cineol on each fabric. On cotton, 1,8-cineol had an absorbance of 0,318 at 1018,44 cm⁻¹, while on polyester, it had an absorbance of 0,170 at 1015,86 cm⁻¹. On the other hand, the blended fabric of polyester/cotton achieved 1016,62 cm⁻¹ (0,282) on 80% polyester, where 65% polyester gained 1016,52 cm⁻¹ (0,222). The summary of the peaks of 1,8 cineol is tabulated in Table 2.

Sample ID	Wavelength (cm ⁻¹)	Absorbance
COT	1018,44	0,318
PE	1015,86	0,170
TYB	106,62	0,282
NKT	1016,62	0,222

Table 2. Wavenumbers and absorbance of 1,8cineol found on samples

For the measurement of eucalyptus oil in this study, it was also observed on FTIR ATR, as depicted in Figure 2. The 1,8 cineol was also shown at 1015,10 cm⁻¹ with the absorbance of 0,464. Figure 2 also represents the eucalyptus oil peaks obtained with an α -pinene instead of β -pinene. According to Amilia *et al.* (2015) and Nuritasari *et al.* (2014), the peak of α -pinene was 2916,37 cm⁻¹ which is aligned with this study result.^{38,39} On the other hand, the peak of β -pinene should show a sharp carbonyl absorption at 1740 cm⁻¹ and an OH absorption at 3590 cm⁻¹, which is not found in this experimental result.⁴⁰



Figure 2. FTIR mapping for eucalyptus oil (*Melaleuca leucadendra* L.) from Perhutani Gundih, Central Java

It was found that the main peak of eucalyptus oil in a specific plant of *Melaleuca leucadendra L*. from Perhutani Gundih has α -pinene with a wavelength of 2917,48 cm⁻¹ at an absorbance of 0,174. The fabric was then analyzed for the difference between before and after treatment, or control and treated samples. Figure 3(a) depicts a sample of control cotton.

The two figures above in Figure 3 point out that one of the eucalyptus oil compounds, α -pinene, is present on the fabric since the wavelength was matched. Furthermore, the other fabrics were also observed, which polyester results are shown in Figure 4.

Like cotton, polyester also had a new peak after the treatment of eucalyptol microcapsules, and the peak was at 2919,03 cm⁻¹ at 0,0399 absorbance. The Toyobo® fabric, which contains 80%/20%



Figure 3. The difference between the control and treated cotton sample with (a) no sharp peak and (b) an apparent new sharp peak with a wavelength of 2917,48 cm⁻¹ at 0,0784 absorbance.



Figure 4. The existence of α -pinene in a treated sample of polyester is shown in (b), while the control sample was (a)

polyester/cotton fibers, obtained the presence of α -pinene. It is shown in Figure 5 below, where a wavelength of 2918,60 cm⁻¹ rises at 0,0593 absorbance.

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Figure 5. The new peak of 2918,60 cm⁻¹ appears on the treated Toyobo® sample (b), which indicates the presence of α pinene, while the control sample (a) has no sharp peak at that specific wavelength

The Nikita® sample, on the other hand, which consists of 65%/35% polyester/cotton, achieved the same change in shifting peak as other treated fabrics did. Figure 6 (a) and (b) below show that the new peak was at a wavelength of 2917,06 cm⁻¹ with an absorbance of 0,0551.



Figure 6. Similar to other treated fabrics, the peak on treated Nikita® also shifts to a number of 2917,06 cm⁻¹

Based on Figures 3 to 6, the existence of α -pinene can be summarized in Table 3.

 Table 3. Wavenumbers and absorbance of αpinene found on samples

Sample ID	Wavelength (cm ⁻¹)	Absorbance
COT	2917,48	0,0784
PE	2919,03	0,0399
TYB	2918,60	0,0593
NKT	2917,06	0,0551

According to Table 3, the results of FTIR ATR for four types of fabric concluded that the compound of eucalyptus oil detected on treated samples was α -pinene, for which the average wavelength was 2919 cm⁻¹.

Antibacterial Analysis

Table 4 below shows the results of antibacterial tests with the cultivating *S. Aureus* bacteria test (according to SNI ISO 20743-2011).

Table 4. Antibacterial test with bacterial inhibitory
values for the treated fabrics with
microencapsulation of eucalyptus oil

Sample ID	Diameter of bacterial inhibition (mm)
COT	19,45
PE	0,00
TYB	17,64
NKT	17,66

Based on Table 4, it can be analyzed that cotton has a higher number of inhibitory values, even though the numbers were not significant. The higher the content of cotton, the greater the effectiveness of antibacterial finishes. The diameter of the samples was 16.64 mm. The difference between the diameter and inhibitory value was 2,74 mm on 100% cotton, while Nikita®, which has 35% cotton, had a difference of 1,02 mm to the diameter of the sample and inhibition diameter zone. Nevertheless, Toyobo® has a 1,00 mm difference. On the contrary, 100% polyester has a 0 mm inhibition diameter zone, meaning the antibacterial finish was not well coated on the fabric. In conclusion, the difference was only less than 5 mm, it determines that the antibacterial finish worked but not at the maximum values. According to the standard of antibacterial tests on textiles, the antibacterial activity worked well if the inhibition diameter zone was more than 5.0 mm, and it was classified as strongly effective if it was more than 10,0 mm.41,42

SEM Analysis

The SEM analysis of the treated fabrics is shown in Figure 7. The presence of small objects on the fabric, suspected as capsules of eucalyptus oil in the gum acacia walls, was analyzed. Figure 7 (a)









Figure 7. SEM photographs of treated fabrics on (a) cotton 100%, (b) cotton 35%, (c) cotton 20% and (d) cotton 0% with 2000x magnification

depicts a COT made of 100% cotton with microcapsules in the interstices of the fibers at a magnification of 2000x and a diameter of approximately 3,75 µm. But the microcapsules did not evenly spread out over the surface. Similar to COT, the NKT's 35% cotton presents small objects on the fabric surface with an uneven spread and shape of prickles, which were also suspected as capsules of finish substance (Fig. 7b). The fabric of TYB, which has 20% cotton, shows the microcapsules but less than COT and TYB, with an approximate diameter of 2,25 µm (Fig 7c). On the other hand, PE that has no cotton fibers presents the least appearance of microcapsules at a magnification of 2000x (Fig. 7d). The chunk of a small object, which is also suspected to be a finished substance, is approximately 5,70 µm in diameter.

CONCLUSION

Four types of fabric (100% cotton, 100% polyester, 80/20 polyester/cotton, and 65/35 polyester cotton) were prepared to have antibacterial finishing properties, and the results were analyzed. The main antibacterial agent was eucalyptus oil, obtained from the leaves of Melaleuca leucadendra L. cultivated in Perhutani Gundih, Central Java, Indonesia. The samples were treated with the oil in the form of microencapsulation. They were then observed on FTIR and antibacterial tests according to SNI ISO 20743:2011. The results show that four types of fabrics were detected as having a compound of eucalyptus oil, α -pinene. For the antibacterial tests, it was found that fabric of 100% polyester has zero diameters of inhibitor area, while the longest diameter was found on a 100% cotton fabric with a 19,44 mm diameter of inhibitory area, compared to a 16,64 mm diameter of samples. Nikita® then followed it with 35% cotton and Toyobo with 20% cotton, which both had 17,66 mm and 17,64 mm diameters of inhibitor field, respectively. A future will be conducted to analyze study the microencapsulation characteristics and wash durability of an antibacterial finish using eucalyptus oil.

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