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Formulation and Evaluation of Halal Cosmetic Product Deodorant Spray from Lignin Nanoparticle of Palm Kernel Shell

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ABSTRACT
Halal cosmetics, including the use of deodorants, have gained substantial attention due to their adherence to ethical, religious, and health-related standards. The lignin extracted from palm oil shells possesses antimicrobial properties, making it a promising candidate for deodorant formulation. This study aimed to evaluate a deodorant formulation incorporating nanoparticle of palm shell lignin and to analyze it as raw material of halal cosmetic. The lignin was synthesized and characterized prior to its incorporation into the deodorant base. The formulated product was then evaluated for its physicochemical properties such as pH, homogeneity, and spreadability. Results showed that palm shell particles exhibited a lignin wavelength of 0.275 nm, an average particle size of 1000 nanometers, and physical properties suitable for cosmetic products. While the evaluation results were promising, further research is needed to optimize the formulation. Variables that could be modified include lignin concentration, type and quantity of additives, and manufacturing method. Palm lignin nanoparticles are a viable and promising material as a raw ingredient for halal cosmetics.

Keywords: Halal cosmetic; palm oil shell; deodorant; nanoparticle; lignin

INTRODUCTION

Halal cosmetics have become a highly relevant issue with the growing awareness of the importance of halal products among consumers. Halal cosmetics are not only required to be free from *haram* (forbidden) or *naji*s (impure) substances but also need to be produced through processes that align with Islamic principles. In Islam, the use of materials that are considered haram or doubtful is believed to lead to a lack of blessings in everyday life (Ahmad et al. 2023). Halal cosmetics emphasize natural ingredients that are safe for health and in accordance with religious teachings. In Indonesia, the demand for halal cosmetics has increased as Muslim consumers seek products that are not only effective but also align with their religious beliefs. This demand presents a significant opportunity to develop cosmetic products based on natural ingredients, such as lignin extract.

Lignin is a natural polymer and is classified as a compound that is difficult to decompose in nature, recalcitrant compound, because it is resistant to degradation or does not degrade quickly in the environment. Lignin molecules are complex organic polymer compounds found in plant cell walls and function to provide strength to plants (Ridho et al. 2022). Lignin is composed of 3 types of phenylpropanoid compounds, namely coumaryl alcohol, coniferyl alcohol and sinapyl alcohol. The most abundant and important polymeric organic substance in the plant world besides cellulose is lignin. Lignin is found in cell walls and some is found in the middle lamella, in the area between cells. The structure of lignin varies greatly depending on the type of plant. In general, lignin polymers are composed of phenyl propane units, namely pcoumaryl alcohol, coniferyl alcohol, and sinapyl alcohol, which are the parent compounds (precipients) of lignin. Physically, lignin is amorphous, bright yellow in color with a specific gravity ranging from 1.3-1.4 depending on the lignin source (Zhang, Haque, and Naebe 2021). The refractive index of lignin is 1.6. Because of its amorphous nature, lignin is difficult to analyze using X-rays. Lignin is also insoluble in water, in acid solutions and hydrocarbon solutions. Because lignin is insoluble in 72% sulfuric acid, this property is often used for testing. The use of lignin as an active ingredient in halal cosmetics not only meets safety and efficacy standards but also supports sustainability and environmental conservation. Lignin, as a natural ingredient, is biodegradable, making it more eco-friendly compared to synthetic chemicals commonly used in conventional cosmetic products.

The palm oil industry is one of the most significant sectors for Indonesia's economy. Riau is recorded as having an oil palm plantation area of 3.38 million hectares or 20.68 percent of the total area of oil palm plantations in Indonesia. In 2022, Riau province will have an area of 2.87 million hectares or 18.70 percent of the total area of oil palm plantations in Indonesia. Meanwhile, community-managed oil palm plantations reach 1.7 million hectares (BPS 2022a). Palm oil plantation commodities have quite an important role in economic activities in Riau because of the high potential to produce palm oil for the industrial sector (Ilhami, Hidayat, and Riandi 2024). The largest production of palm oil (CPO) in 2022 will come from Riau Province with production of 8.74 million tons or around 18.67 percent of Indonesia's total production which is supplied from smallholder plantations (55.13%), state plantations (4.5%) and private plantations (40.36%) (BPS 2022b).

The large amount of palm biomass produced includes palm kernel shells (Pulingam et al. 2022). Palm oil shells (*Elaeis guineensis Jacq.*) contain lignocellulosic compounds which are polysaccharide components. Lignocellulose consists of three types of polymer, namely 13-28% cellulose, 21-22% hemicellulose, and 44-52% lignin (9)(10). Lignin, as the highest component, has been widely researched and applied in various fields. Lignin has been considered as a potential replacement for fossil fuels in the development of new materials or chemicals due to its low price, biodegradable, biocompatible, non-toxic properties and its use as a carrier for various anticancer drugs (Chaudhary and Sinha 2023; Schneider, Dillon, and Camassola 2021). Lignin can be used in biofuels (diesel, synthesis gas, hydrogen, charcoal); chemicals (dispersants, binders, polymers); agriculture (controlled release of pesticides and herbicides, heavy metal adsorbing agents, as a component of humic acid formation in the soil structure and as a high-value fertilizer); human health (drug encapsulation and delivery, treatment of obesity, diabetes, thrombosis, viruses, and cancer); as well as other fields such as UV protective cosmetics, cement and batteries. Additionally, due to its antioxidant and antimicrobial potential, lignin has been explored as an ingredient in various other diseases such as diabetes, orthopedic diseases, and inflammatory bowel diseases (Schneider et al. 2021).

Currently, there has been a lot of research that utilizes palm oil waste, such as research used activated palm shell charcoal as a halal beauty treatment for the skin (Edo et al. 2022). Making soap from palm kernel oil (Muhammed et al. 2022) and solid soap formulation combining activated palm shell charcoal and sodium lauryl sulfate (Coiffard and Couteau 2020). Other research includes the formulation and stability test of hair tonic preparations from oil palm leaf extract in protecting hair (Lestari and Farid 2023) and the formulation and evaluation of the wound healing activity of oil palm leaves. Previous research also proves that lignin has potential as a main ingredient in biomedical applications because it has various benefits, including as an antioxidant, antibacterial, and anti-Ultraviolet (UV), as well as showing positive biodegradability and biocompatibility properties (Lalouckova et al. 2021).

The development of science and technology provides solutions related to delivery systems for cosmetic preparations, namely in the form of nanoparticles, because the large size of lignin particles causes the effectiveness of preparations on the skin to decrease. So the use of nanoparticle technology is an effective solution in the delivery system for cosmetic preparations (Khan and Gupta 2023; Zhou et al. 2021). Previous studies have confirmed that lignin silver nanoparticles (LNPs) can be used as antioxidants, UV protectants in sunscreens, antimicrobials, drug carrier systems for gene delivery, for encapsulation of hydrophobic molecules, for tissue engineering, for hybrid nano-composites as biocatalysts, for immobilization enzymes, for dye removal, for food packaging, for wound dressings, as supercapacitors, as nanotraps for flocculation, and for heavy metal adsorption (Kumar et al. 2021; Piccinino et al. 2021; Tang et al. 2020) . Apart from that, the use of lignin from palm oil shells will help reduce industrial waste and is of course environmentally friendly because lignin can decompose naturally. The aim of this research is to formulate Lignin nanoparticles by utilizing palm shell biomass as a deodorant spray preparation and to analyze its suitability as a raw material for halal cosmetics.

METHODOLOGY

Materials

Kraft lignin form palm kernel shell (Riau, Provinci), AgNO3 (*Merck milipore*), NaOH 20% (*Merck milipore*), H2SO4 20% (*Merck milipore),* Aquabidest (*Bratachem*), Propilen glikol (*Bratachem*), PEG 400 (*Bratachem*).

Instrumentation

Spektrofotometri UV-1900i (Shimadzu), Particle Size Analyzer (Horiba), X-Ray Diffraction X'Pert PRO (PANanalytical, Almelo, Belanda).

Characterization of Lignin

Spectro UV-vis

UV–vis spectra of lignin were recorded on a spectrometer (UV-1900i, Shimadzu) in the range of 200 – 800 nm. The deionised water was used as a blank sample. Three measurements were performed for each sample, and its average value was presented in the graphs.

Particle size distribution

Lignin nanoparticles were examined using the dynamic scattering (DLS) method with Horiba (Horiba Instruments). For zeta potential measurements, the Smoluchowski model is applied to electrophoretic mobility. All measurements were performed in triplicate.

X-Ray Diffraction

The crystal structure and crystallite size of freeze-dried Lig-NPs samples were determined using X-ray diffraction (XRD) with a PANalytical X'Pert PRO instrument (PANanalytical, Almelo, The Netherlands). Powder diffraction data were collected at room temperature using an X'Pert3 θ-θ powder diffractometer with Bragg–Brentan parabolic focusing geometry and CuKα radiation (λ = 1.54060 Å, U = 40 kV, I = 30 mA). The data was scanned in the angular range of 5°–90° (2θ) with a step size of 0.0390° (2θ) and a calculation time of 354.96 s $step^{-1}$. PIXCEL ultrafast 1D detector was used for data collection. Data evaluation was carried out using HighScore Plus 4.0 software (Malvern Panalytical, Malvern, UK) and the PDF-4+ database as a reference.

Formulation of deodorant spray

Procedure for Making Deodorant Spray Nanoparticles Spray is made using a simple solution method. First, a co-solvent system was created by dissolving silver nanoparticles in water and stirring until the solution was homogeneous. Then other solvents such as propylene glycol and PEG 400 were added and stirred thoroughly. Oral spray is made by adding other additives for good taste, good physical characteristics and stability. The ingredients of the oral spray are shown in Table 1.

Table 1. Ingredient Formulation for Deodorant Sprays

Ingredient	Formula (g)
Propilen glikol	15
PEG 400	40
Parfum	qs
Aquadest	Ad 100

RESULT AND DISCUSSION

Isolation and Lignin characterization

Isolation of lignin from palm shell residue was carried out using the klason method with prehydrolysis and delignification process stages (Sangthong et al. 2022). The prehydrolysis process is the first stage in cooking which aims to weaken the bond between hemicellulose and the lignin component by adding NaOH until the simplicia looks crushed and softened. This is because NaOH is able to degrade lignin compounds and penetrate into the simplicia and weaken the lignin bond with hemicellulose with additional heating so that the lignin can be separated from the hemicellulose (Pongchaiphol et al. 2022)

After the prehydrolysis process is carried out, it is then continued with the Delignification process, where this process aims to separate the bonds of the lignin components with hemicellulose by adding H_2SO_4. This process is carried out to ensure that the lignin bond with hemicellulose can be separated so that only lignin compounds are expected to be obtained. Then, from isolation using the klason method, lignin yield results were obtained. These results show that the klason method is able to extract lignin compounds from palm shell simplicia.

The lignin characterization carried out was determining the pH, determining the wavelength, determining the spectrum, determining the methoxyl content and determining the molecular weight. The pH determination was carried out using a pH meter to determine the pH of the lignin, the pH determination result obtained was 7.6. This is because the lignin has gone through a washing process using distilled water until it is free from acid. Then the wavelength is determined using a UV-Vis spectrophotometer with the principle of interaction between light and matter so that the wavelength of the lignin is obtained. The result obtained is that the lignin wavelength is 0.275 nm. Apart from determining the wavelength, the spectrum of lignin was also determined using an IR spectrophotometer, the results obtained were 3320 cm-1. These results are obtained from light and matter based on functional groups.

The results of particle size analysis using PSA show that the successfully synthesized lignin nanoparticles have an average size of 1000 nanometers. This relatively non-uniform particle size indicates that there is a synthesis process in producing nanoparticles with a controlled size. The narrow particle size distribution also shows that the majority of particles have sizes that are not much different from the average. This small particle size is very important because it provides a large surface area, thereby increasing the potential for nanoparticle interactions with polymer matrices or other compounds in subsequent applications.

XRD analysis provides information regarding the crystal structure of lignin nanoparticles. The increase in the crystallinity index of lignin nanoparticles compared to the original lignin indicates a change in the lignin molecular structure due to the nanoparticle formation process. This can be caused by several factors, such as the breaking of certain chemical bonds, reorganization of the lignin polymer chains, or the formation of new crystal structures. This change in crystal structure can affect the physical and chemical properties of lignin nanoparticles, such as solubility, thermal stability, and the ability to interact with other compounds. The presented XRD spectrum provides an interesting insight into the crystallographic structure of lignin. Sharp Diffraction Peaks show that there are a large number of sharp diffraction peaks indicating the presence of a fairly good crystal structure in the lignin sample. This contradicts the common perception that lignin has an amorphous structure. The complex diffraction pattern indicates the presence of multiple crystalline phases or a possible mixture of amorphous and crystalline phases in the sample. The label on the diffraction peak indicates the presence of the Na_2SO_4 phase. This indicates contamination or addition of these compounds during the sample preparation process. Although it is difficult to identify with certainty the peaks originating from pure lignin, some unlabeled peaks may represent contributions from the lignin crystal structure. However, this contribution is relatively small compared to the contribution from the Na_2SO_4 phase. The width of the diffraction peaks can be used to estimate the size of the crystallites. Sharp peaks indicate relatively large crystallite sizes. However, more accurate calculations require the use of special XRD analysis software. The complex diffraction patterns indicate that the lignin crystal structure may have a high degree of order. However, further analysis using crystal structure databases is needed to identify specific lignin crystal phases

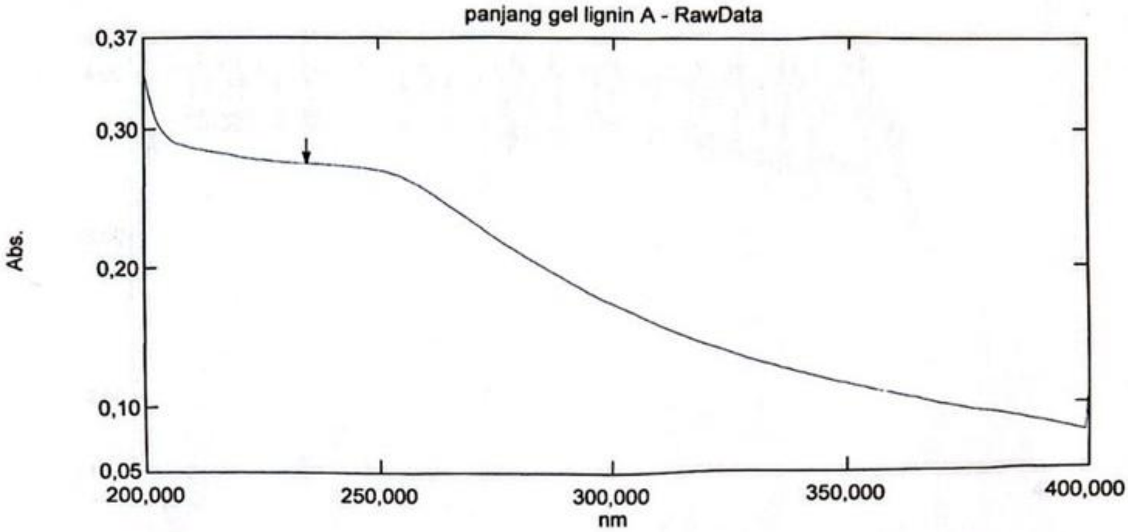


Figure 1. Lignin wavelength using spectro uv vis

Zeta potential analysis was carried out to evaluate the stability of the resulting lignin nanoparticle dispersion. The measurement results show an average zeta potential value of 0.7 mV at a temperature of 25°C, with the dispersion medium having a viscosity of 0.896 mPa.s, conductivity of 16,519 mS/cm, and electrode voltage of 1.3 V. The zeta potential value obtained indicates that the interactions between lignin nanoparticles tend to be weak. The zeta potential value that approaches the isoelectric point indicates the potential for aggregation or precipitation of particles in the dispersion. Weak interactions between particles can be caused by several factors, including the presence of functional groups on the particle surface which are not sufficient to provide strong electrostatic repulsion, as well as the possibility of dominant van der Waals forces. The zeta potential value obtained was 0.7 mV indicating that the surface of the lignin nanoparticles was positively charged. This relatively small surface charge causes attractive forces between the particles, resulting in an unstable nanoparticle dispersion in solution for a long time. A zeta potential value that is close to zero indicates that particles tend to clump or settle. To increase the stability of the palm shell nanoparticle dispersion, surfactants were added. Surfactants have hydrophilic and hydrophobic groups. The hydrophilic group will interact with the solvent (for example water), while the hydrophobic group will interact with the particle surface. Thus, surfactants can form a layer on the surface of the particles, increase the surface charge, and reduce the attractive forces between particles. Good dispersion stability is very important to prevent agglomeration or settling of particles, which can reduce the specific surface area and reduce the performance of nanoparticles in applications.

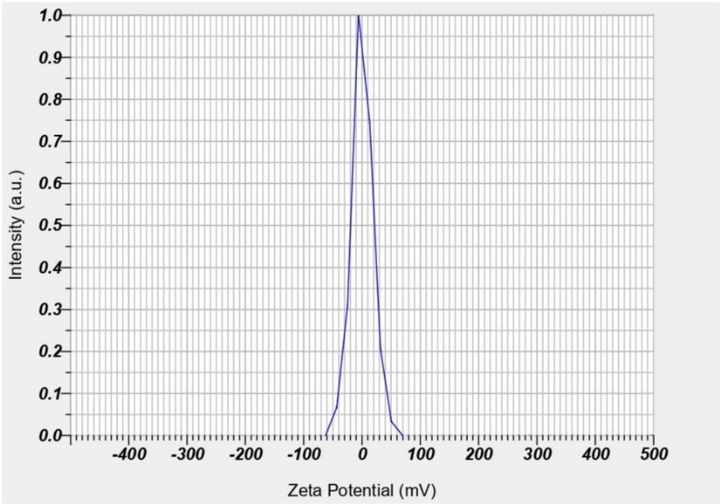


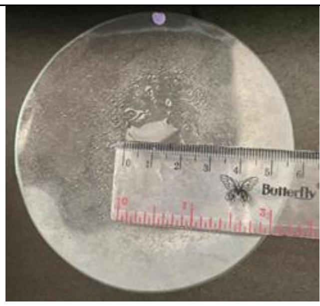
Figure 2. Zeta potential analyis of shell palm oil

Analysis of the characterization of palm shell lignin using the PSA (Particle Size Analyzer) and XRD (X-ray Diffraction) techniques provides in-depth insight into the potential use of lignin as an active ingredient in deodorant formulations. The results of PSA analysis show that lignin particles have a very favorable size because they provide a large surface area, thereby increasing the potential for lignin interaction with skin and microorganisms that cause body odor. The lignin crystal structure obtained from XRD analysis shows the presence of hydrophilic and hydrophobic functional groups that have the potential to interact with sweat and sebum components in the skin. Hydrophilic groups can help lignin be adsorbed on the skin surface, while hydrophobic groups can bind molecules that cause body odor. Apart from that, the unique crystal structure of lignin can also provide natural antibacterial properties, so it can inhibit the growth of bacteria that cause body odor. Based on previous research results, lignin has been reported to have good antioxidant and antimicrobial activity. These properties are very relevant to the function of deodorants, namely to inhibit bacterial growth and reduce the production of odorous compounds. The potential of lignin as an active deodorant ingredient is supported by its unique physicochemical properties, such as a large surface area, complex crystal structure, and the presence of reactive functional groups (Mondal et al, 2023).

Evaluation of deodorant spray using lignin nanoparticle

Palm nanoparticles, derived from palm oil industry waste, have attracted attention as potential active ingredients in deodorant formulations. The unique nanoparticle structure with a large surface area allows more effective interaction with body odor molecules (Kankala et al. 2020). In addition, the bioactive compounds contained in palm nanoparticles, such as flavonoids and phenols, have antibacterial and antioxidant properties which can inhibit the growth of bacteria that cause body odor and neutralize odorous compounds. In formulating palm nanoparticle-based deodorants, several important considerations need to be taken into account. First, the stability of the nanoparticle dispersion in the deodorant base must be ensured. The use of surfactants or polymers can help increase dispersion stability and prevent particle agglomeration (Cortés et al. 2021). Second, you also need to pay attention to the particle size of the nanoparticles, because particles that are too large can clog skin pores. Apart from that, the safety of using palm nanoparticles on the skin also needs to be tested extensively to ensure there are no adverse side effects.

Table 2. Evaluation of deodorant spray using lignin nanoparticle

Evaluation Parameter	Description	Result
Spreadability test	The existing palm shell antiperspirant deodorant is placed or smeared on a scaled glass, then the top part is given the same glass, and the load is increased, and given a time span of 1-2 minutes. Then calculate the diameter of the spread.	 Figure 3. Spreadibility test
Homogenity test	This test is carried out by looking at the preparation with the naked eye, whether there are coarse particles or not, if not then the preparation is said to be homogeneous.	 Figure 4. Homogenity test
pH test	The pH test is carried out by taking 1 strip of litmus paper, then dipping it in the bubble deodorant test solution where the pH level will be measured, then waiting for 5-10 seconds, then lifting the litmus paper and matching the color change to the existing indicator. on litmus paper packaging containers	 Figure 5. pH test

Evaluation of palm shell lignin-based deodorant formulations has been carried out by measuring several important parameters, namely pH, spreadability and homogeneity. The results of this evaluation provide an overview of the quality and potential of the deodorant formulation being developed. The pH value of the deodorant formulation obtained is within the range that is considered safe for the skin. An appropriate pH will help maintain the skin's natural pH balance and minimize the risk of irritation. These results indicate that the developed formulation has the potential to be used as a personal care product. Good spreadability of a deodorant formulation means that the product is easy to apply to the skin and provides broad coverage. The spreadability test results show that the lignin-based deodorant formulation has quite good spreadability. This indicates that the product can provide effective protection against body odor. Formulation homogeneity is an important factor influencing product quality. A homogeneous formulation will provide a more attractive appearance and ensure even distribution of active ingredients. The test results show that the deodorant formulation developed has a good level of homogeneity. This indicates that all components in the formulation are evenly dispersed, so they can provide optimal performance. The evaluation results obtained indicate that palm shell lignin has potential as an active ingredient in deodorant formulations. Lignin is known to have antibacterial properties which can help inhibit the growth of bacteria that cause body odor (Lie et al, 2023). Apart from that, lignin is also biodegradable, so it is more environmentally friendly than synthetic materials. Although the evaluation results show quite good results, further research needs to be done to optimize the formulation. Several parameters that can be varied include lignin concentration, type and amount of additional ingredients, and manufacturing method. In addition, it is necessary to test its effectiveness against bacteria that cause body odor to confirm its claim as a deodorant.

Palm Lignin Nanoparticles as Raw Material for Halal Cosmetics

In Islam, "Halal" refers to that which is permitted or allowed to be consumed according to Islamic law (Salamon et al, 2021), as well as to the teachings of Prophet Muhammad. As stated in Surah Al-Maidah: 4 of the Qur'an:

"They ask you (Muhammad), 'What has been made lawful for them?' Say, 'Lawful for you are (all) good things and (the animals of) prey which you have trained for hunting, in the manner taught you by Allah. So eat of what they catch for you and mention the name of Allah upon it, and fear Allah. Verily, Allah is swift in reckoning"
(Al-Maidah: 4)

This verse explicitly clarifies what is considered lawful in Islam. Halal, as mentioned in the Qur'an, refers to what is permissible or allowed in Islam. Everything in this world is halal unless there is a prohibition explicitly outlined. Ramadan (2024) defines halal as a product or service recognized by the authority, in accordance with Sharia law, and carries the halal logo. Therefore, the term 'Halal' is not limited to the ingredients alone but extends to the processes of production, storage, packaging, and distribution of products, all in compliance with Sharia law. The halal industry can encompass all consumer goods, including pharmaceuticals, cosmetics.

Halal cosmetics are products that must not contain human body parts or ingredients derived from animals that are prohibited for Muslims; they must not contain genetically modified organisms (GMOs) that are deemed impure (najis); they must not contain alcohol (khamr); there should be no contamination from impurities during preparation, processing, manufacturing, and storage; and they must be safe for consumers (Nordin et al., 2021). Furthermore, the use of animal fats and testing, hazardous chemicals, and other substances considered unacceptable by Muslim consumers is prohibited . All halal beauty products are also recognized as clean, safe, and high-quality products. As narrated by Daruquthni and authenticated by Imam Nawawi, the Hadiths of the Prophet Muhammad regarding the permissibility and prohibition of certain consumables

"Allah has enjoined certain obligations; do not neglect them. He has set boundaries; do not transgress them. He has prohibited certain things; do not violate them. He has left some matters unspecified out of mercy toward you, not due to forgetfulness. Therefore, do not inquire about the rulings on those matters"
(Reported by Daruquthni and authenticated by Imam Nawawi)

Lignin is a natural compound found in plant cell walls, particularly in palm oil shells. Lignin possesses antimicrobial, antioxidant, and biodegradability properties that make it highly suitable for application in the halal cosmetics industry. These properties allow lignin to combat bacteria responsible for body odor, protect the skin from damage caused by free radicals, and be environmentally friendly due to its ability to decompose naturally. Therefore, lignin offers a natural alternative that is not only safe for health but also supports environmental sustainability (Luzi et al, 2021).

Lignin nanoparticles extracted from palm oil shells hold great potential as raw materials for halal cosmetics. In addition to providing benefits from its active properties, lignin obtained from palm oil waste is a cheap and sustainable resource. The utilization of palm oil shells as a lignin raw material can reduce the environmental impact caused by the abundant waste from the palm oil industry (Uchegbulam,2022). Moreover, the use of this natural material supports the principles of sustainability and the circular economy, which are increasingly emphasized in the global cosmetics industry. Lignin is a phenolic compound found in plant cell walls, which naturally does not contain haram ingredients such as alcohol or animal fats. In the production of palm lignin nanoparticles, there are no processing or additive ingredients involving haram components, ensuring that cosmetic products containing palm lignin nanoparticles meet halal criteria.

Palm lignin nanoparticles are a viable and promising material for halal cosmetics. From the perspective of resource availability, safety, skin health benefits, and alignment with Sharia principles, palm lignin nanoparticles offer an innovative and environmentally friendly solution for the halal cosmetics industry. By emphasizing natural quality and skin-friendliness, while avoiding haram ingredients, cosmetic products based on palm lignin nanoparticles not only meet the needs of halal cosmetics but also provide optimal health benefits for their users (Suryanegara, 2024). According to the fatwa of the Indonesian Ulama Council (2018), the use of cosmetics for the purpose of adornment is permissible under the condition that: the ingredients used are halal and pure; intended for purposes that are permitted by Islamic law; and do not pose any harm

CONCLUSION

The results of this research indicate that lignin extracted from palm shells has promising characteristics for application in cosmetic products. Lignin's wavelength of 0.275 nm indicates a unique molecular structure that can provide certain functional properties. The average particle size of 1000 nanometers and other physical properties that comply with cosmetic standards indicate the potential of palm shell lignin as an additive or carrier in various cosmetic formulations. Further research is needed to optimize the lignin extraction and characterization process, as well as evaluate its effectiveness in cosmetic products in vivoterisasi lignin, and evaluate its effectiveness in halal cosmetic products in vivo.

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