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RESEARCH ARTICLE

Formulation and Characterization of *Sertu* Kaolin Clay Paste for Cleaning of Medical Equipment and Wound Treatment

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Abstract The treatment of the wounds inflicted by dogs and wild boars becoming a significant public concern. From Islamic contexts, it demands effective and Shariah-compliant cleaning methods. Current practices predominantly employ soil-based soap, raising questions about its scientific efficacy and safety for wound treatment and medical equipment cleansing. This study is executed to formulate and characterize a Shariah-compliant kaolin clay paste designed for *sertu* applications in the medical field, for both wound areas and medical equipment cleansing. Kaolin clay was selected as the soil medium in the formulated *sertu* clay paste and underwent various characterizations, including X-ray Diffraction (XRD), Scanning Electron Microscope (SEM) and Fourier Transform Infrared (FTIR) analyses. XRD analysis confirmed the presence of kaolinite in the kaolin clay. This is also further evidenced by the presence of bonds between Al and Mg ions and hydroxyl groups in the FTIR spectra. The irregular morphology of the kaolin particles observed through SEM, contributed to an increase in surface area, thereby enhancing the interaction between clay and other components. The formulated clay paste was further evaluated through pH measurement, irritation analysis, antimicrobial testing, and corrosion testing to assess its performance. The results indicated that the formulation produced a stable *sertu* clay paste, with pH values ranging from 8.34 to 9.52 and a primary irritation index (PII) of 4.38 which is within acceptable range for wound treatment. Additionally, the paste proved suitable for cleansing medical instruments, as no significant corrosion or bacterial growth was observed.

Keywords: Sertu, FTIR, irritation analysis, corrosion analysis, kaolin clay.

Introduction

Animal bites due to dog attacks and wild boar are common problems in Malaysia between 2017 – 2020, a total 71,045 dog and animal bites cases were reported [1]. Beyond the immediate physical trauma, these bites can cause severe and even deadly infections if not treated promptly because of the potential of spreading harmful bacteria into the victim's body. According to Meyer [2], injuries from dogs and wild boar can lead to a serious infection that is known as toxemia and improper treatment could cause rabies infection. Mannion and Graham [3] have addressed that dog bite injuries frequently occur on the head and facial area for children whereas in adults, the most commonly affected areas are the wrist and hand. They also mentioned that dog bites can result in the appearance of puncture wounds and crush injuries. Puncture wounds pose a high risk of infection due to the difficulty in thoroughly cleaning the affected area. Morgan and Palmer [4] reported that the infected wounds of injury are usually due to the transmission of *Pasteurella multocida*. Antimicrobials are usually used to prevent the spread of this bacteria [4].

This issue is multifaceted, as the cases referred to hospitals require meticulous attention to wound treatment from the bites and it's essential to note that the treatment process poses challenges for medical equipment as well, since the saliva from the bites is considered impure (*najs mughallazah*) in Islamic jurisprudence (*fiqh*) [5]. In the discussion of Islamic jurisprudence (*fiqh*), particularly in Madhhab al-

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Shafi'i, "*sertu*" is a method of purifying impurities from dogs and boars. Formulating a Shariah-compliant *sertu* requires adhering to specific principles and procedures outlined in Islamic jurisprudence (*fiqh)*. The core components of a Shariah-compliant *sertu* formulation include the primary medium of water mixed with soil (*turbatun)* (clay or earth). Even though no specific type of soil is mentioned in the book of Fiqh, the type of soil and its purity is needed to be considered for hygienic cleaning and the proportion of soil to water are crucial aspects for the shariah compliance. This mixture is used to cleanse the affected body part or object. Types of clay ordinarily used in *sertu* purification include Kaolin Clay, Bentonite Clay, Fuller's Earth, Red Clay, White Clay (China clay), and local clays based on traditions [6 – 8]. For Muslims, it is compulsory to apply the *sertu* procedure to clean any areas that are bitten by dogs or attacked by wild boars. Other than that, any equipment that encounters *najs mughallazah* also needs to be cleaned accordingly based on the same procedure. Due to the beneficial properties of certain soils and clays—such as kaolin, which has traditionally been used in skincare, it is a great advantage to use it as the main gradient in *sertu* composition.

Kaolin is primarily composed of the mineral kaolinite, which is a hydrated aluminium silicate [$Al_2(Si_2O_5)(OH)_4$], which stands out among clay minerals due to its remarkable properties. Kaolin consists of tiny sheets of triclinic crystals with pseudohexagonal morphology. It is formed by rock weathering and has some cation-exchange capacity as well as possessing low viscosity and stability [9]. The kaolinite group is classified as a 1:1 type layer silicate with a tetrahedral sheet of silica (SiO_2) joined together with an oxygen atom and an octahedral sheet of alumina (Al_2O_3). Kaolinite possesses high chemical stability, low expansion, and cation exchange capacity. The kaolinite group is structurally divided into dioctahedral and trioctahedral minerals [10]. Its fine particle size, typically ranging from 0.2 to 2 micrometres, grants kaolin a smooth texture and a platy or pseudohexagonal shape. Naturally white or light in colour, it exhibits chemical inertness and thermal stability, even at high temperatures. While not as plastic as some clay minerals, kaolin retains moldability when mixed with water, possesses low viscosity, and has good solution stability. Additionally, its absorbent nature makes it apt for moisture control in various applications, including reducing the corrosive effects on metals. These characteristics render kaolin suitable for wound and medical equipment cleansing,ensuring effective contaminant removal while maintaining hygiene in compliance with Shariah principles.

The current use of soil-based soap for medical equipment and wound treatment lacks scientific support, posing safety risks raising safety concerns. Ensuring patient well-being and Shariah compliance requires a thorough understanding of the properties of the clay used in the *sertu* process. Developing the correct emulsion or paste for wound care is imperative. The absence of clear scientific Islamic guidelines for dog bite treatments highlights the significance of this research, which focuses on formulating and characterizing *sertu* clay paste for medical equipment cleansing and wound treatment.

Materials and Methods

Materials

Kaolin clay, ethylenediaminetetraacetic acid (EDTA) as a chelating agent, paraben as a preservative, and sodium laureth sulfate (SLES) as a surfactant were sourced from a local Malaysian supplier, Morning Prestige Trading. The project was structured into distinct phases, including formulation and characterization.

Formulation of *Sertu* Kaolin Clay Paste

The clay paste was prepared by blending kaolin clay, distilled water, EDTA, paraben, and SLES according to the proportions detailed in Table 1.

Characterization of the Kaolin Clay and Formulation Paste

The analyses conducted to assess the properties of the raw materials, specifically kaolin, included X-ray diffraction analysis (XRD), morphological examination, and functional group assessment. The XRD pattern of kaolin was examined using a Bruker D2 Phaser X-ray Diffractometer operating at 40 kV and 20 mA. The kaolin was scanned from 10^{O} to 50^{O} (2Θ) with a scan rate of 37s/step.

Table 1. Formulation of the kaolin clay paste

Formula	Water (ml)	EDTA (g)	Paraben (g)	Kaolin clay (g)	SLES (g)
1	60	1	4.5	24.5	10
2	300	5	22.5	122.5	50
3	300	5	22.5	7.5	50
4	300	5	22.5	10	50
5	300	5	22.5	15	50
6	300	5	22.5	30	50
7	300	5	22.5	30	100
8	300	5	22.5	30	150
9	300	5	22.5	100	50
10	320	5	22.5	100	72.5
11	270	17.5	50	110	72.5
12	325	17.5	50	110	72.5
13	300	10	20	100	70
14	300	12.5	35	100	62.5
15	300	7.5	30	100	62.5

Morphological features of the raw materials were visualized through Scanning Electron Microscope (SEM) (JEOL, JSM-5600) operating at 7kV, while the functional group analysis was conducted employing Fourier Transform Infra-Red (FTIR) spectroscopy (Perkin Elmer 100 Series), characterized by a 4 cm^{-1} resolution and 32 scans within the spectral range of 1000 - 4000 cm^{-1}.

The formulated paste underwent various characterization methods, including pH value measurement, assessment of antimicrobial properties, skin irritation test, and corrosion property evaluation. The pH value was determined following the ASTM D4972-19 standard using a pH meter.

The antimicrobial properties of the *sertu* paste were assayed using the agar diffusion method. The microbe strain of Gram-negative *Escherichia coli* (E.coli) and Gram-positive *Staphylococcus aureus* (S. aureus) were used. The microbial suspension was standardized to inoculate petri dishes containing LB agar for bacteria. 6 mm diameter disks were placed on the inoculated agar then at 37°C for 24 h. The diameter of growth inhibition zones around the disk was gauged visually. The antimicrobial activity was expressed as the mean of the inhibition diameters (mm).

The *sertu* solution was tested in an animal irritation test and conducted according to ISO 10993-10. Three healthy male rabbits of New Zealand white strain with weight range of 4.43 - 5.18 kg were used. About 24 hr before the test, the dorsal area of the rabbit was shaved 10 cm x 15 cm at four different sites. The *sertu* solution was applied to the two sites, a negative control site was applied with normal saline while a positive control site with sodium laureth sulfate (SLS)(20% concentration). These sites were individually covered with double layer surgical gauze and wrapped with an adhesive tape for 5 hr After that all patches were removed, and the applied sites were wiped with moist cotton wool to remove any residual. Observations were made for 1, 24, 48 and 72 hr after the removal of test patches. The reactions which are defined as erythema (E) and oedema (O) were evaluated according to the skin reactions scoring system.

The corrosion behaviour of implants and medical instruments like surgery scissors (Figure 1) in different *sertu* solutions was determined using the weight loss method. The samples were cut into a small size of about 0.5 cm, weighted on an analytical balance, and immersed for 1, 2, and 3 months in a five-*sertu* formulation of F5-F10. During the interval, the samples were removed from the *sertu* solution, washed with distilled water, dried, and then weighted again. The equation of weight loss percentage is calculated using Eq. (1)

$$Weight\ loss\ (\%) \quad = \frac{W_o - W_f}{W_o} x100 \tag{1}$$

where W_o is the initial mass and W_f is the final mass.

Figure 1. Medical instrument for corrosion testing

Results and Discussion

X-Ray Diffraction (XRD) Analysis of Kaolin Clay

XRD analysis was performed to identify the presence of mineral species in kaolin and its phases. Based on the XRD spectra pattern in Figure 2, the kaolinite is evident with the presence of absorbance peaks at 2θ = 12.30°, 20.64°, 21.34°, 24.78°, 35.90°, and 38.44°. This observation aligns with the findings of Kumar and Lingfa [11], who reported absorption peaks ranging from 12.34° to 38.46° for kaolin clay. Notably, another higher crystalline peak occurs at an absorption peak of 24.78°. Absorption peaks at 35.90° and 38.44° are indicative of the presence of a mineral known as quartz (SiO_2). Quartz is a common mineral that often appears in XRD patterns at around this angle [11].

Abdelnaby *et al*. [12] suggested that the peak near 35.74° may be attributed to silicon oxide compounds. Silicon oxide compounds can encompass a range of minerals, including various forms of quartz and other silicate minerals. The exact mineral responsible for this peak would require further analysis and comparison to known reference patterns. The XRD analysis provides compelling evidence of kaolinite's presence within the kaolin clay sample, a fundamental criterion for its suitability in *sertu* purification rituals adhering to Islamic jurisprudence. In the context of medical applications such as wound treatment and medical equipment cleansing, knowledge of the clay's mineral composition assumes significance. Kaolin's inherent chemical inertness and absence of reactive minerals make it an ideal choice for contact with the human body [13] and medical instruments. This analysis assures the stability of clay and absence of harmful substances, endorsing its reliability in maintaining cleanliness and safety during medical procedures and rituals.

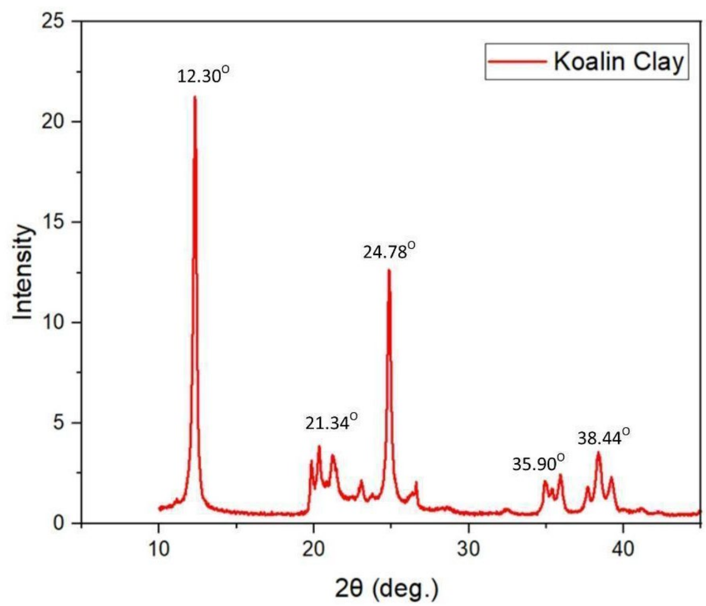

Figure 2. XRD pattern of raw kaolin clay

Fourier Transform Infrared Spectroscopy (FTIR) of Kaolin Clay

The functional group analysis of kaolin clay was examined by using FTIR analysis. The spectrum and corresponding band assignments are shown and tabulated in Figure 3 and Table 2. It can be observed that three prominent bands can be detected at 3687 cm^{-1}, and 3619 cm^{-1}, signifying the O-H stretching region's presence. These bands align with the typical O-H stretching patterns, indicative of the hydroxyl groups in kaolin clay, as noted by Wang *et al*. (2022) [14]. On the other hand, Si-O bonds, a characteristic of kaolin clay, appeared within the 1000 – 1114 cm^{-1} range, attributed to SiO_4 molecules [15]. Symmetric stretching of Si-O bonds was evident at 786 cm^{-1}, 748 cm^{-1}, 699 cm^{-1}, 639 cm^{-1}, and 604 cm^{-1}. Additionally, the 798 cm^{-1} peak was associated with Al-Mg-OH vibrations of hydroxyl groups. The bond between Al and Mg ions and hydroxyl groups is an intrinsic feature of kaolin clay. The presence of these ions and their bonding with hydroxyl groups contributes to the clay's chemical stability. It ensures that the clay remains inert and does not undergo undesirable chemical reactions when it comes into contact with other substances, including impurities or contaminants. The hydroxyl groups are associated with the material's ability to interact with water and other polar substances, which is important in preparing the *sertu* paste. They enable clay particles to bind through hydrogen bonding, ensuring structural integrity. By regulating hydroxyl-containing compound concentrations, viscosity is controlled, facilitating easy application. Uniform dispersion of clay particles prevents clumping for consistent cleansing results. Hydroxyl groups enhance absorbency by forming hydrogen bonds with water molecules, vital in regulating liquid absorption in applications such as wound treatment and cleaning fluids. Their chemical compatibility with clay minerals like kaolinite ensures stability and prevents undesirable reactions, guaranteeing the *sertu* paste's effectiveness during wound care and medical equipment cleansing.

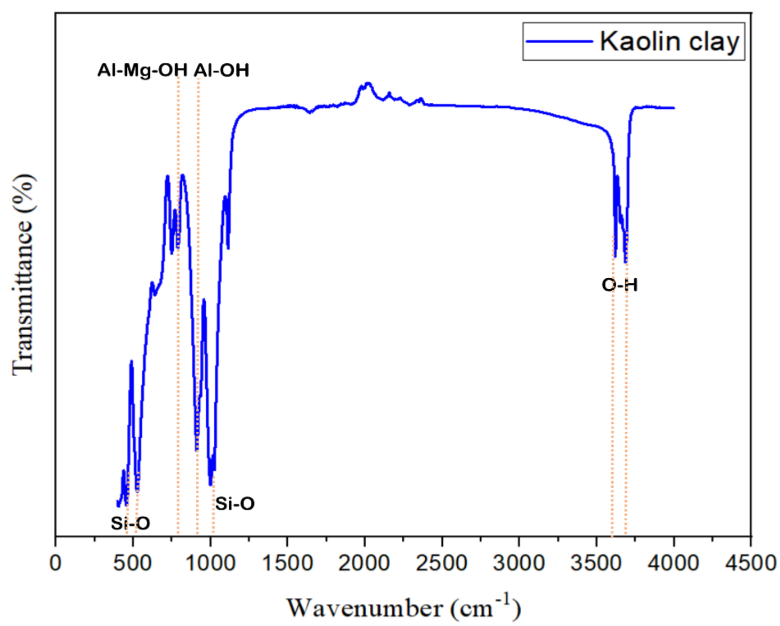

Figure 3. FTIR spectrum of kaolin clay

Table 2. FTIR bands of kaolin clay with their possible assignments

No.	Band (cm^{-1})	Assignments
1	3687 and 3619	O – H (structural hydroxyl group)
2	1114, 1024 and 1000	Si – O (Si – O bonds in the SiO4 molecule)
3	939, 911	Al – OH vibration
4	798	Al – Mg – OH
5	786 and 748	Si – O symmetric stretching
6	699, 639 and 604	Si – O symmetric stretching
7	526, 503	Si – O
8	456, 405	Si – O

Morphology Properties of Kaolin Clay

Figure 4 presents the micrograph of kaolin clay captured using a scanning electron microscope (SEM). The microstructure of kaolin clay, as revealed through SEM analysis, plays a crucial role in shaping the formulation of *sertu* paste. The micrograph shows that kaolin clay exhibits an irregular or plate-like structure, a characteristic also observed in a study by Ababneh *et al*. [16]. The irregular morphology of kaolin particles observed in the micrograph contributes to the increment of surface area and enhances the interaction between the clay and the surrounding medium, a fundamental factor in determining the paste's effectiveness in cleansing procedures. Additionally, Figure 4 highlights the presence of agglomerations approximately 10 µm in size, which necessitate specific dispersion techniques or the incorporation of suitable additives to ensure uniform particle distribution within the paste. Proper dispersion is crucial for achieving consistent and reliable results in *sertu* and cleansing applications. The irregular shape of kaolin particles may impact their ability to bind together and form a stable paste. Understanding this microstructure aids in optimizing the paste formulation to achieve the desired consistency, cohesiveness, and overall performance.

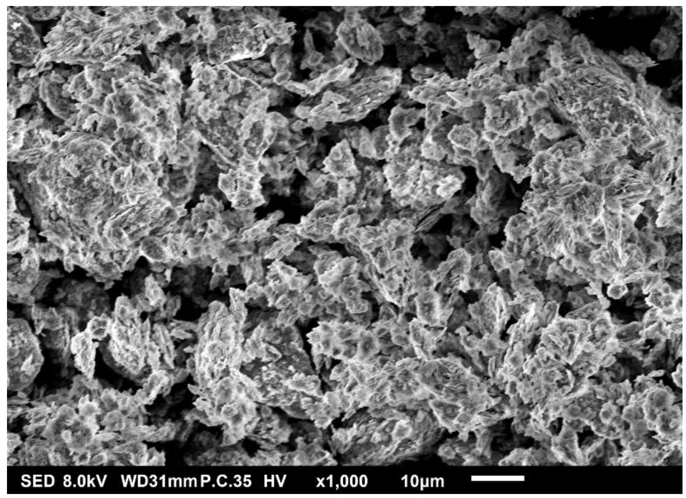

Figure 4. SEM micrograph of raw kaolin clay

Formulation of Kaolin Clay Paste And pH Measurement

The stability of the *sertu* clay paste formulation was assessed over six months, revealing variations among the samples. As shown in Figure 5, samples F12–F15 formed homogeneous mixtures. In contrast, samples F1–F11 exhibited phase separation, with sedimentation at the bottom (some samples are not shown here). This instability was attributed to insufficient interaction between kaolin clay and other ingredients, particularly SLES, which plays a crucial role in reducing surface tension and facilitating micelle formation. The study highlighted the importance of balancing SLES, clay content, EDTA, and paraben to achieve formulation stability.

From Figure 6, pH measurements indicated that samples F10–F15 exhibited higher alkalinity (pH 9.31–9.36) compared to samples F1–F9 (pH 8.34–8.51), suggesting a potential correlation between pH and irritation response. Additionally, pH influences EDTA's ability to chelate divalent cations, with its chelating capacity increasing exponentially between pH 8 and 10 [17]. Similarly, parabens are effective as preservatives in acidic, neutral, and mildly alkaline conditions but lose their efficacy at pH levels above 8 [18].

In this study, although samples F8–F10 had not yet achieved full stability, they were nevertheless tested for antimicrobial efficacy as a preliminary assessment. Stability was not a critical factor at this stage, as antimicrobial effectiveness was expected to remain consistent across similar compositions. The primary objective was to confirm the antimicrobial potential of the *sertu* clay paste, regardless of its stability at that point. Sample F13, formulated later with improved stability, was prioritized for irritation testing. Since skin irritation is influenced by formulation stability, pH, and ingredient interactions, it was necessary to use an optimized sample. Thus, selecting samples F8–F10 for antimicrobial testing and sample F13 for irritation assessment ensured a comprehensive evaluation of both functional and safety aspects of the *sertu* clay paste while addressing stability concerns at different testing stages.

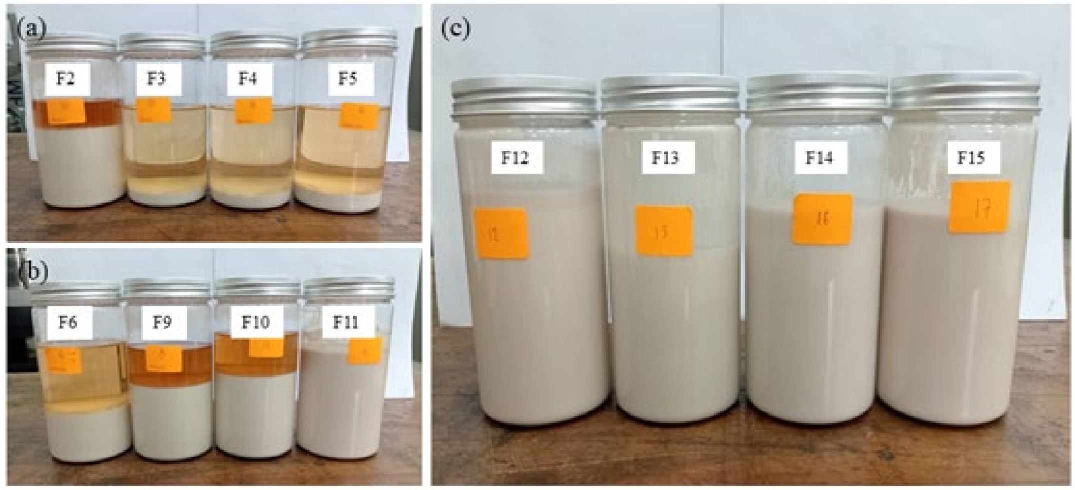

Figure 5. Stability of formulated kaolin clay paste after 6 months

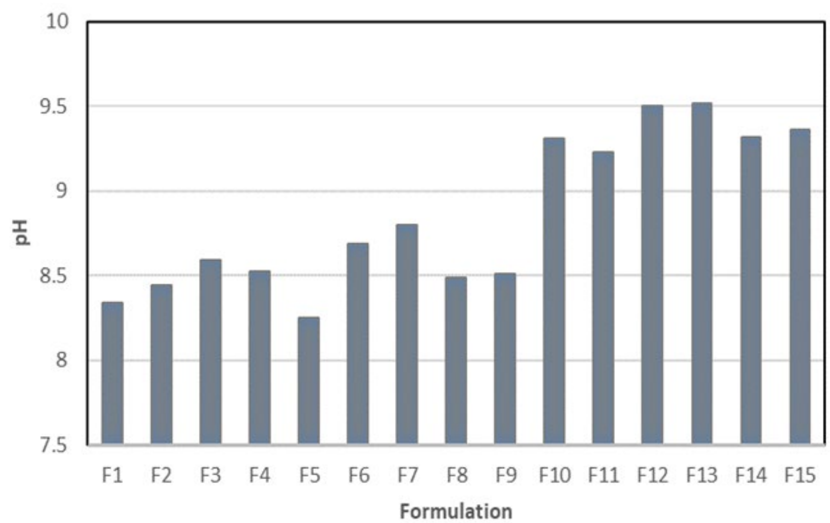

Figure 6. pH value of formulated kaolin clay paste

Irritation Response of Formulated Kaolin Clay Paste

To evaluate the effect of formulated kaolin clay pastes on users, a skin irritation test was conducted. Sample F13 was selected for the irritation response test due to its superior stability and well-balanced formulation. The tested animals were monitored for behavior and activity levels. According to the report, the rabbits appeared active and healthy, showing no signs of gross toxicity or abnormal behavior. Based on the irritation score in Table 3, the formulated *sertu* clay paste (F13) falls into the moderate category, with a primary irritation index (PII) of 4.39. This indicates that the *sertu* clay paste has the potential to induce skin irritation upon application. It is believed that the high pH value of the paste contributes to this irritation, as suggested by Hawkins *et al*. [19], who reported that higher pH levels in skin products can lead to an increased irritation index, thereby causing skin irritation. As mentioned in the methodology section, the test was conducted over 72 hours. However, in Shariah practices, the *sertu* solution is required only for a single application to cleanse the wound, followed by rinsing with water six times. Given this short exposure duration, the likelihood of the *sertu* solution causing significant skin irritation is extremely low.

Based on pH values in Figure 6, although samples F12 and F13 had similar pH levels, differences in ingredient concentrations likely influenced their long-term stability. Sample F12 contained higher amounts of EDTA (17.5 g) and paraben (50 g) compared to sample F13, which had 10 g of EDTA and 20 g of paraben. While EDTA helps prevent degradation as a chelating agent, excessive amounts may affect formulation consistency over time. Similarly, parabens act as preservatives, but high concentrations can alter texture and stability. Sample F13 was selected for its superior stability, maintaining a uniform mixture with minimal phase separation. Its slightly lower SLES content (70 g vs. 72.5 g in F12) may have contributed to better surfactant balance, preventing excessive micelle formation that could impact consistency. Given these factors, sample F13 was prioritized for irritation testing, ensuring a stable, effective *sertu* clay paste with optimal microbial resistance and user safety.

Table 3. Primary irritation index for *sertu* solution and normal saline under 72 hours observation period

	Primary Irritation Score (Mean)	
Animal number	Test Item (Sertu Solution)	Negative control (Normal Saline)
AR00N (male)	5.50	0
AR003N (male)	6.17	0
AR002M (male)	2.83	0
Primary Irritation Index	4.89	0

Antimicrobial of Formulated Kaolin Clay Paste

The antimicrobial properties of the formulated *sertu* clay paste against *E. coli* and *S. aureus* (Figure 7) were evaluated using the disc diffusion method by analyzing inhibition activity. The inhibition activity was measured based on the size of the inhibition zone. Figure 7 presents the *sertu* clay paste samples F8, F9, and F10 (as labeled) after 24 hours of incubation. The inhibition activity of other formulations is not shown here, as no changes were observed in their inhibition zone size. The absence of inhibition zones indicates that bacteria did not proliferate within the formulated *sertu* clay paste. As reported by Azmi *et al*. [20], the absence of inhibition zones signifies a lack of bacterial growth in the sample.

As seen in Figure 7, most of the *sertu* clay formulation showed efficient inhibition of both bacteria. However, S. aureus (Gram-positive) appears to be more susceptible to *sertu* clay paste, in which a slightly larger inhibition zone is noticed in sample F10 compared to other samples. Clay, including kaolin, has been reported to possess antimicrobial activity [21], primarily due to its metal complexes and cations. This structure allows it to attract toxins and absorb pathogens. Additionally, the antimicrobial properties observed in this study may also be attributed to the presence of EDTA and paraben. EDTA functions as a chelating agent, binding to iron, which is a crucial element for microbial metabolism and growth [22]. Meanwhile, paraben exhibits strong antimicrobial activity, particularly against fungi and Gram-positive bacteria [23], which aligns with the findings of this work. Hence, these findings underscore the antimicrobial effectiveness of the clay paste, with implications for its utility in cleansing and *sertu* procedures, as well as its potential benefits in wound care.

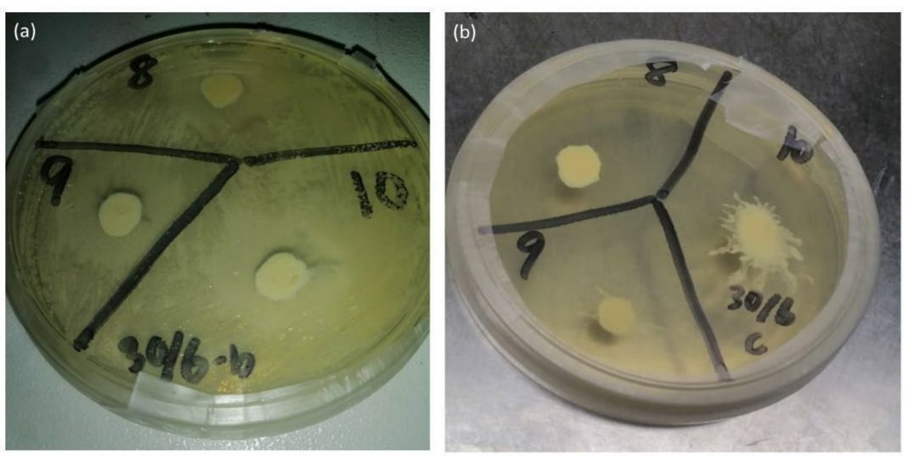

Figure 7. *Sertu* clay paste in sample (a) E. coli, and (b) S. aureus, after 24 hours

Corrosion Analysis of Formulated Kaolin Clay Paste

Figure 8 shows the weight variations of samples F5-F8 throughout three months. The formulations were chosen to represent alkaline pastes for corrosion analyses as the pH of the samples are within the range of 8-9. It is noted that the alkaline pH range does not promote electrochemical reactions leading to corrosion. The weight loss equation was not used due to the fact that no significant changes in weight were observed. Remarkably, samples F5, F6, F8 and F9 did not undergo weight reduction, signifying that the formulated *sertu* clay pastes did not induce corrosion product deposition on these medical instrument samples. In the case of sample F7, a minor weight reduction of approximately 3% (0.31 g) was observed in the second month, but no further decrease in weight occurred in the next third month. This suggests that the corrosion mechanism did not accelerate further for sample F7. An alkaline *sertu* paste might prevent corrosion by promoting passivation or forming protective oxide layers on metal surfaces. It is known that the medical instruments are made from the high-grade stainless steel, nevertheless, prolonged exposure to some chemical and environmental conditions could also cause corrosion. From this study, the consistent weight of the medical instrument samples implies that the *sertu* clay paste is a suitable cleansing agent for medical instruments, as it does not promote corrosion.

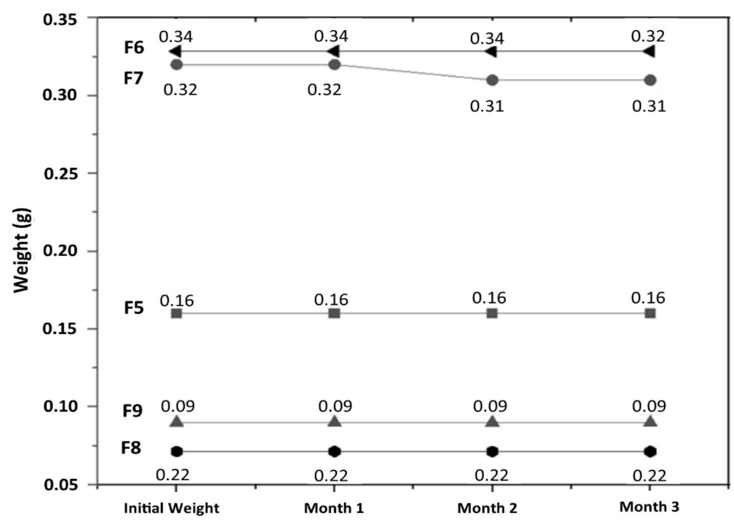

Figure 8. The weight of five kaolin clay paste tested samples

Conclusions

This research on formulated kaolin clay paste for *sertu* applications concludes that the kaolin clay used meets the purity and stability requirements set for the *sertu* process. The unique microstructure of clay characterized by irregular shapes and porosity, significantly influences its properties such as surface area, absorbency, dispersion, and binding. With a moderately alkaline pH, the clay paste of formulation F13 is suitable for the purpose and its moderate irritation results may also be suitable for a single wash cleaning with this paste before rinsing with clean water as in the guideline of the *sertu* process. The paste exhibits antimicrobial properties, as indicated by the absence of bacterial growth in the inhibition zone, making it valuable for maintaining cleanliness during purification and cleansing. Additionally, it does not induce corrosion in stainless steel, confirming its suitability for cleansing medical equipment. While it may cause irritation when applied to wounds, the skin irritation test affirms its safety for cleaning medical instruments.

Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper.

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