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# Compositional Engineering of Chitosan–Potato Starch–Kaolin Biocomposites: Tuning Physicochemical Properties for Wound Dressing Applications

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**Abstract**

An investigation has been made to predict the effects of various Kaolin fillers with different concentrations from 4 to 10 percent on the properties of Chitosan and Potato Starch Bio-composite for wound dressing applications. The procedure was to prepare the chitosan and starch in equal proportions, apply the filler to the hydrogel, dry it, and characterize it. The characterizations of the synthesized bio-composite were its absorption percentage, mechanical test, presented functional groups, and antibacterial test. It was found out that the bio-composite with the highest swelling percentage 6% Kaolin, showed 128.65% swelling. Moreover, the highest percentage of bio-composite absorption was 65.62% with 8% Kaolin added. In addition to that, adding 4% Kaolin produced the highest tensile strength with the value of 75.6 MPa. Besides, the most significant elongation percentage was 151.60% in the addition of 4% Kaolin. Qualitatively, the synthesized bio-composites exhibited functional groups of OH, alkynes, and carboxylic acid. Finally, the bio-composite with Kaolin filler significantly performed in antibacterial activities.

***Keywords***: *Bio-composite, Chitosan, Kaolin, Potato Starch, Wound Dressing.*

## 1. Introduction

Applying a wound dressing can help the wounded skin heal. Wounded areas can be dressed with bandages that promote healing. Wound dressing pads must keep the body steady, regulate excessive exudate, establish a moist environment, and resist microbial transfer. Making biodegradable wound dressings is one attempt to solve the wound-healing issue. Among the primary components of biodegradables is starch, an inexpensive, readily obtainable hydrocolloid that comes in various forms in Indonesia. Starch and chitosan combination advancement as a raw wound dressing material has increased [1].

A naturally existing polymer with antibacterial, biocompatible, and wound-healing qualities is chitosan. Potato starch is a cheap, readily available, naturally occurring substance that is easily broken down. The prospective use of these two polymers as wound dressings has not yet been thoroughly investigated. We can produce a material with improved performance and biocompatibility by mixing chitosan with potato starch in a bio-composite [2]. Potato starch is not a newly used material as a bio-composite. It has been used as a promising material in making bio-composites with its outstanding performance of oxygen barrier and water vapor [3].

Conventional or synthetic wound dressings often fail to offer the best possible care and protection for wounds. Consequently, it's critical to design novel and potent wound dressing materials. The chitosan derived from shrimp shell chitin possesses antibacterial, biocompatible, and biodegradable qualities. One naturally occurring starch source with strong biodegradability is potato starch [4]. However, adding kaolin to these bio-composites can overcome the shortcomings of chitosan and potato starch's mechanical strength and stability.

Primary wound dressings are typically composite materials covered in a thin layer that acts as a barrier against bacteria. Other requirements for primary wound dressings include being elastic, having high absorbency, preventing bacterial attacks, sealing the wound, and retaining moisture to prevent damage to newly formed tissue. The primary prerequisites for bio-medical polymers are that they should be non-toxic, allergy-free, easily sterilizable, possess sufficient mechanical qualities, be robust, elastic, long-lasting, biocompatible, and derived from easily obtainable biomaterials that require little processing. Additionally, they should have antimicrobial qualities and the ability to heal wounds [5].

This study aims to develop a wound dressing with strong antibacterial capabilities, superior mechanical strength, and the ability to absorb exudate (wound fluid) by mixing chitosan, potato starch, and kaolin. If this objective is met, the wound dressing will promote healing, stop infections, and expedite healing.

## 2. Literature Review

In the present research, the bio-composite fillers were Kaolin ($Al_2O_3.2SiO_2.2H_2O$). One mineral that is frequently found in the Earth's crust is Kaolin. Kaolin is a type of rock that is primarily white or somewhat whitish and is formed of clay material with a low iron content. In addition to treating adult cases of moderate diarrhoea, it is also used as a lotion for abrasions, boils, and skin inflammation; however, antiseptic and antibiotic creams have mostly taken its place. These two materials are also easily decomposed by the environment, so they can reduce the negative impact on the environment if used as wound dressings [6].

### 2.1. Biocomposite

Biocomposites are a sustainable option that can replace standard synthetic composites. They are environmentally friendly, light, and can break down naturally, making them useful in industries like cars, building, textile, pulp and paper, packaging, and medicine, such as wound dressing[7] [8][9]. Making biocomposites means mixing natural fibers or fillers with bio-based or synthetic polymers. Many studies have shown that natural fibers are better than artificial materials like glass or carbon fibers. Natural fibers can be regrown, are not heavy, and break down naturally. They also have pretty good strength and stiffness [10]. However, they tend to soak up a lot of water, which can cause them to stick poorly to other materials and change shape [11].

### 2.2. Chitosan

Chitosan is a natural sugar that comes from chitin. Chitin is found in the shells of shellfish and the cell walls of fungi. Chitosan has become popular because it works well with living tissues, breaks down naturally, is not poisonous, and has many bodily uses [12]. Its special physical and chemical features make it worthwhile for medicine, the environment, farming, and food packaging. Many studies have shown that chitosan can kill a wide range of bacteria and fungi [13]. The amino groups, which have a positive charge, connect with the membranes of microbial cells and break them down. Chitosan's ability to fight oxidation comes from its ability to eliminate free radicals, which is helpful in medicine and keeping food fresh.

### 2.3. Wound Dressing

Wound dressings are essential for treating both new and long-lasting wounds. The primary purpose of these dressings is to help wounds heal, stop infections, handle any fluids coming from the wound, and create the best conditions for new tissue to grow. Wound dressings have changed a lot over time, from basic gauze and bandages to special materials and active dressings designed for different kinds of wounds and stages of healing[14].

## 3. Methods

This study used glassware, a vacuum oven, a centrifuge (Hettich type EBA 20), and a glass Mold (7cm x 7cm x 5mm). The following equipment was used to characterize the bio-composite: anti-bacterial, swelling, absorption, mechanical, and Fourier-transformed infrared spectroscopy (FTIR) functional group testing.

### 3.1. Materials

The materials utilized in this research were: Chitosan (shrimp shell from PT BCI), glacial acetic acid (Analytical grade 0,5M, VWR, Int.), Kaolin (SIGMA), Glycerol (SIGMA).

### 3.2. Variables

At the chitosan-potato starch hydrogel processing stage, the variable conditions are as follows:

Fixed variables: chitosan from shrimp shell type, potato starch, concentration of chitosan and potato starch in acetic acid: 0.5%, DD of chitosan: 90.2%, concentration of acetic acid: 1%, room temperature drying time: 48 hours, drying temperature 50ºC, variation in chitosan-potato starch ratio (w/w): 50/50. Hot Plate temperature when mixing: 50°C, Hot Plate time when mixing: 20 minutes, Drying temperature: 50°C, Drying time: 6 hours, Glycerol volume: 2 ml, Chitosan mass: 2 grams, Potato starch mass: 10 grams.

Independent variables:

Kaolin filler percentage (w/v): 4,6,8 and 10

Dependent variables: absorption percentage, mechanical test, functional groups, and antibacterial test.

### 3.3. Procedures

This study consisted of four procedures: chitosan and potato starch processing, Kaolin filler application, drying, and examining or testing. The chitosan mixture was poured into the biopolymer mixture with varying masses and mixed. The mixture was then homogenised to attain a well-mixed chitosan and potato starch mixture alongside Kaolin filler. After that, the mixture was centrifuged to separate undissolved parts.

Having achieved a more homogeneous mixture with fewer water bubbles, the mixture was injected into a glass mould 7x7 mm with 5 mm thickness to be a bio-composite. It was then placed at room temperature for 48 hours, followed by another 48 hours drying at 40ºC. The dried bio-composite was then released from the mould and rinsed with distilled water five times before it was re-dried and placed in a cuvette to be further examined. The schematic diagram of the study procedure is shown in Figure 1.

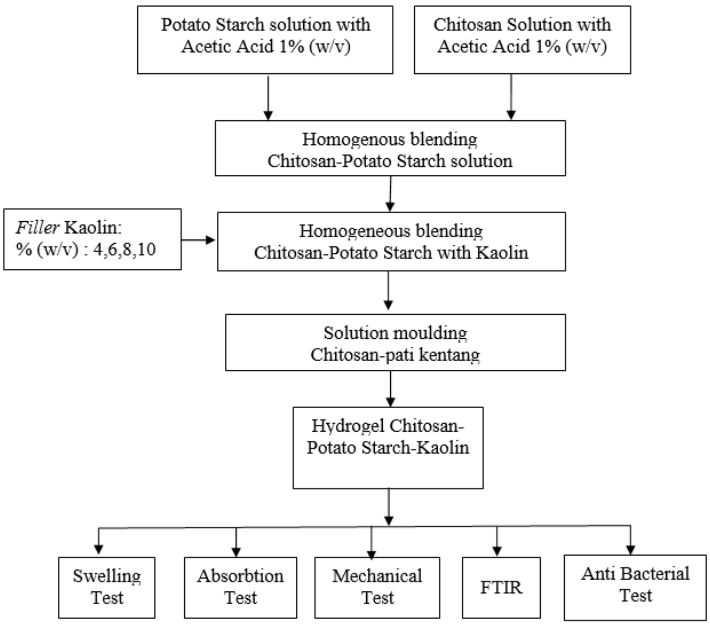



**Fig 1.** Schematic Diagram of Research Procedure

## 4. Results and Discussion

The findings of this research activity include how to process (under what conditions) chitosan-potato starch-based bio-composites for biomedical applications (primary wound dressings), how to produce chitosan-potato starch bio-composite products with the addition of filler of Kaolin, the outcomes of bio-composites' swelling and absorption tests, the results of FTIR functional group tests, the results of mechanical testing, and the results of anti-bacterial tests.

### 4.1. Figure captions

The Chitosan-Potato Starch-Kaolin bio-composites produced can be seen in Figure 2.

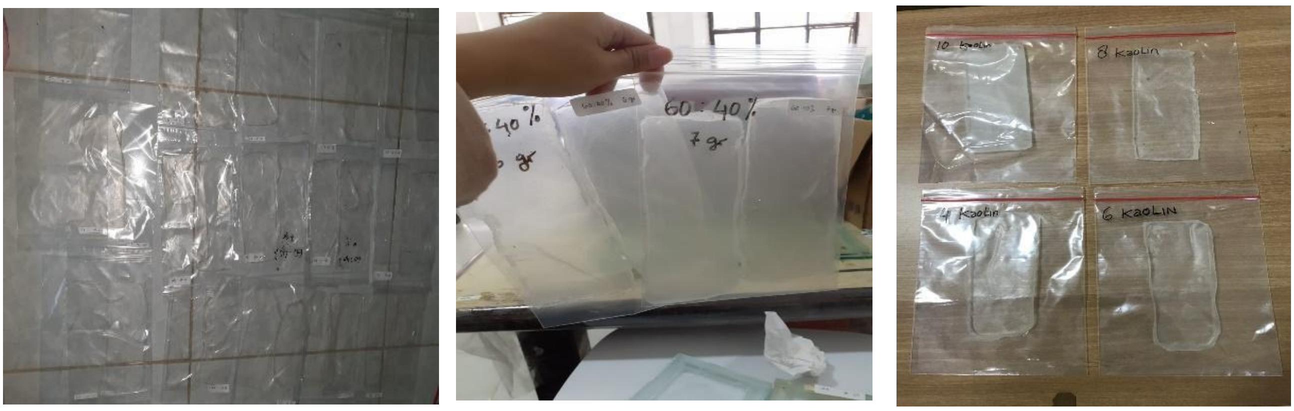



**Fig 2.** Chitosan-Potato starch-Kaolin Biocomposites Film

The bio-composite made of chitosan, potato starch, and kaolin is non-porous, elastic, translucent, and thin-looking. Adding 4-6 grams of Kaolin is more elastic than adding 8–10 grams. Due to their stretchy nature and ability to stick to the wound surface, the modified versions of these two bio-composites are perfect for wound dressings. Patients experience discomfort from stiff, brittle, and dry wound dressings because the ideal wound dressing should be elastic and comfortable to apply and remove.

### 4.2. Swelling Percentage

An assessment of the bio-composite's resistance to absorbing liquid was made using the water resistance test. This determines how much fluid is absorbed, so the bio-composite expands when applied as a wound-covering material. Maintaining moisture in the wound is the best condition for effective wound healing[15]. For each variation, the bio-composite was cut to a two by 2 cm size, and the sample was then submerged in NaCl for four hours after the initial weight was measured. In the swelling test, the fluctuation graph of the bio-composite, chitosan, potato starch, and Kaolin is shown in Figure 3.

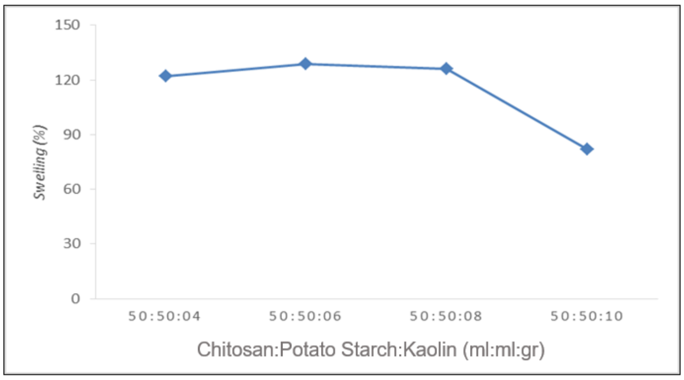



**Fig 3.** Kaolin Correlation with Swelling Percentage

It can be seen from Figure 4 that the percentage of 10% Kaolin is increased, and the swelling value tends to decrease. The most significant percentage swelling value of 128.65% was achieved by combining 6% kaolin with a 50:50 potato starch-chitosan mixture. In the meantime, 10% kaolin produced 82.14% of the lowest percent swelling value for the potato starch-chitosan v/v% (50:50) combination.

## 4.3. Absorption Percentage

The absorption test is intended to ascertain the absorption capacity of potato starch-chitosan and Kaolin bio-composite. The ability of a wound dressing to absorb is one of its key characteristics. This is determined by the fact that the larger the absorption capacity, the more wound exudate it can absorb[16]. Phosphate Buffer Saline (PBS) solution with a pH of 7.3 was the liquid utilized in this experiment for 12 hours. The PBS tablet was dissolved in 100 millilitres of distilled water to create the PBS solution. Weighing the bio-composite before and after soaking allowed us to determine the absorption percentage. Figure 4 illustrates the relation between Kaolin and bio-composite absorption.

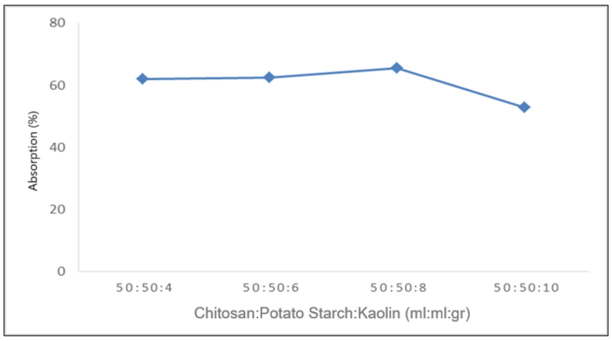



**Fig 4.** Kaolin Correlation with Absorption

It is apparent that as the 10% Kaolin composition increases, the percentage absorption value tends to decrease. The composition of potato starch-chitosan v/v% (50:50) with 8% Kaolin yielded the highest percent absorption value, 65.62%. In the meantime, 10% Kaolin produced the lowest percent swelling value, or 52.81%, for the potato starch-chitosan (50:50) v/v% composition.

## 4.4. Absorption Percentage

Tensile strength is the most significant tensile strength that a bio-composite can have before failing[17]. Every material has various properties (such as flexibility and hardness). Testing is necessary to determine a material's properties. The goal of a stress test, one of the most commonly performed tests, is to ascertain the material's properties and strength level. The matrix and filler significantly impact the tensile strength of composite materials. Whereas the filler is a filling material (fibre), the matrix is a composite binder or protective material. Figure 5 illustrates the effect of Kaolin on bio-composite tensile strength.

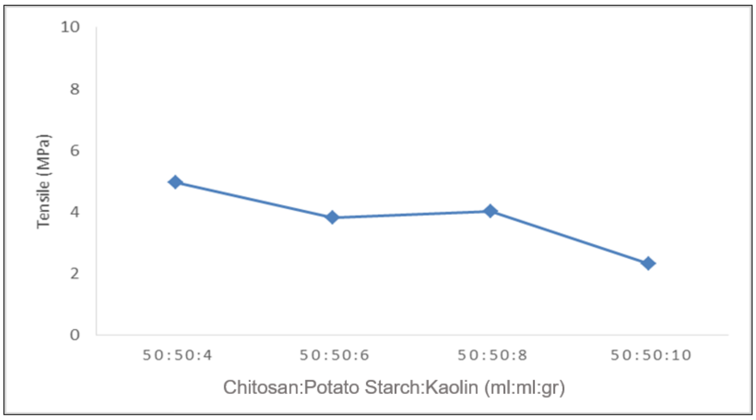



**Fig 5.** Kaolin Correlation with Tensile Strength

As shown in Figure 5, bio-composites typically lost some of their tensile strength as kaolin was introduced. After adding 4 grams of Kaolin, the bio-composite's tensile strength dropped from 4.97 MPa to 2.34 MPa.

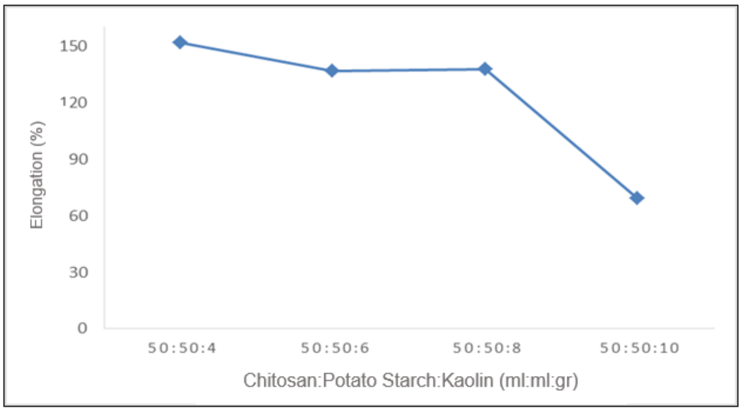



**Fig. 6.** Kaolin Correlation with Elongation Percentage

From Figure 6, it can be seen that the elongation value tends to decrease as the percentage of Kaolin increases. In this case, when 10% Kaolin is incorporated, the elongation decreased from 151.6% to 69.5%. [18]stated that because the resulting bio-composite can withstand specific loads and tensile forces, a high elongation percentage indicates that it does not break easily. Because hydrocolloid1 has a lubricating effect that makes the edible film emulsion more elastic and stronger, using it can increase the breaking strength and elongation percentage.

## 4.5. Existence of Functional Groups (FTIR)

Functional group analysis is used to identify constituent compounds—especially organic ones—both qualitatively and quantitatively. This study utilized chitosan and potato starch as raw materials, and Kaolin was added as a filler. Chitosan is a naturally occurring polymer comprising three functional groups: amino acids, primary, and secondary hydroxyl groups. On the other hand, starch is derived from carbohydrates, specifically glucose. This polymer compound comprises three main components: amylose, amylopectin, and intermediate materials like protein and fat. The resulting analysis of Kaolin variation is shown in Figure 7.

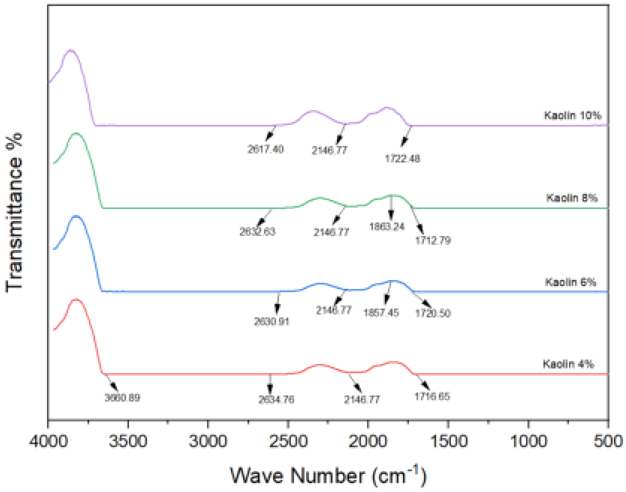



**Fig. 7.** Functional Group Analysis on Chitosan-Potato Starch-Kaolin Bio-composite

Figure 7 displays the compounds and functional groups formed in the membrane layer. Based on these findings, it can be concluded that the sample containing 4% Kaolin included an O-H group at 3660.89 $cm^{-1}$, a C≡C alkyne group at 2146.77 $cm^{-1}$, and C=O (carboxylic acid) at 1716.65 $cm^{-1}$. Table 1 shows the analysis.

**Table 1.** Functional Group Analysis on Chitosan-Potato Starch-Kaolin Bio-composite at Different Wavelengths ($cm^{-1}$)

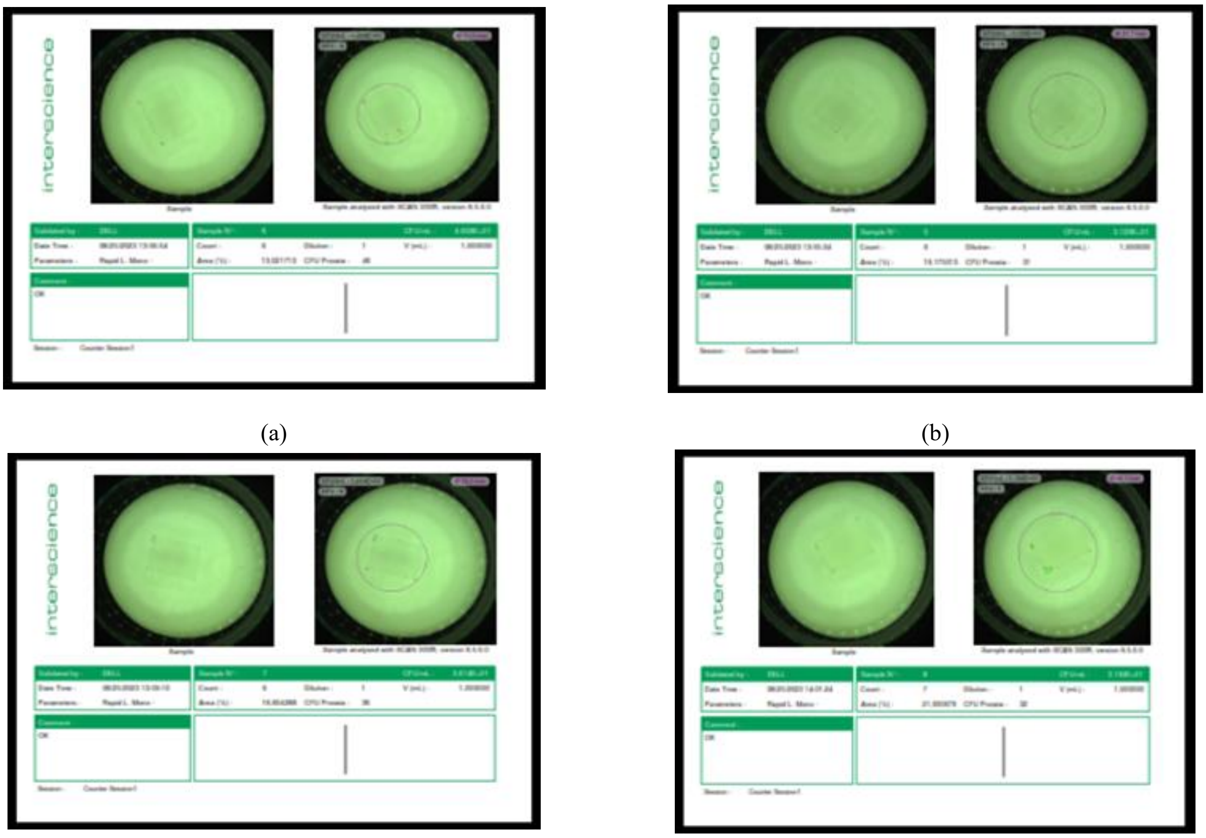
| Functional Groups | Kaolin Composition (%) | | | |
|---|---|---|---|---|
| | 4% | 6% | 8% | 10% |
| C=O Carboxylic Acid | 1716.65 | 1720.5 | 1712.79 | 1722.43 |
| | | 1857.45 | 1863.24 | |
| C≡C Alkynes | 2146.77 | 2146.77 | 2146.77 | 2146.77 |
| O-H /Acidic | 2634.76 | 2630.91 | 2632.83 | 2617.4 |
| O-H Alcohol | 3660.89 | 1720.5 | 1712.79 | 1722.43 |

The bio-composite's functional groups were analysed using a combination of kaolin, potato starch, and chitosan. The results indicate that the compound contains O-H hydroxyl groups derived from starch. Due to its abundance of hydroxyl groups, starch, a polysaccharide in potatoes, is hydrophilic and can form hydrogen bonds with water molecules[19]. In FTIR analysis, the hydroxyl group (O-H) in starch will exhibit a typical absorption at approximately 3300-3600 cm-1. The stretching and bending vibrations of the O-H groups in the starch

molecules are reflected in this absorption peak. This hydroxyl group is also essential in starch's capacity to absorb kaolin and create a complex, creating a bio-composite of both.
As kaolin is added to the potato starch-chitosan bio-composite, the C-H group will typically absorb light at a wavelength of 2850–3000 $cm^{-1}$. If there are any C-H bonds in the molecules of starch, chitosan, and maybe kaolin, this absorption peak represents their C-H stretching vibrations. The presence of C-O and O-H functional groups in the analysis results suggests that the dressing is generally hydrophilic.

## 4.6. Anti-Bacterial Test

The diffusion method is employed in the resistance test against bacteria, and the pathogenic bacteria used are S. aureus and E. coli. The formation of an inhibition zone or clear zone around it indicates the organism's sensitivity to the test sample. It is possible to identify which organisms are susceptible or resistant by comparing the diameter of the clear zone with the standard[20].
The current investigation used the diffusion method with paper discs as the antibacterial test method. The purpose of measuring antibacterial activity is to locate and assess a substance that may have antibacterial activity against bacteria[21]. The Chitosan-Potato Starch-Kaolin Bio-composite antibacterial analysis is shown in Figure 8.



(a) (b) (c) (d)

**Fig. 8.** Antibacterial Analysis on Bio-composite with Varying Kaolin (a) 4% (b) 6%, (c) 8% and (d) 10%

Examining the findings of the antibacterial test analysis from Figure 8 using a composition of 4% kaolin-potato starch-chitosan, 46 bacterial colonies were found, and 32 of those colonies contained 10% kaolin with 7 CFU/mL produced by bio-composite. The broader zone of inhibition created suggests that the chitosan, starch, and kaolin bio-composite can effectively combat bacterial activity.

## 5. Conclusion

The effects of varying fillers and their percentage on bio-composites have been investigated. Some concluding observations from the investigation are given below.

1. The bio-composite of chitosan, potato starch, and kaolin (50:50:6) showed the best composition variation in the swelling analysis with a percentage of 128.65%.
2. The composition variation (50:50:8), or 65.62%, was the best chitosan-potato starch-kaolin bio-composite for the absorption test.
3. The highest mechanical (tensile) test results were 4.97 MPa and 75.6 MPa for chitosan-potato starch-Kaolin (50:50:4).
4. Chitosan-potato starch-Kaolin (50:50:4) had the highest mechanical test results (elongation), at 151.6%.
5. The findings for the chitosan-potato starch-Kaolin bio-composite indicated the presence of the constituent components, which included an O-H group at 3660.89 $cm^{-1}$, a C≡C alkyne group at 2146.77 cm-1, and C=O (carboxylic acid) at 1716.65 $cm^{-1}$.
6. The Chitosan-potato starch-Kaolin (50:50:8) bio-composite produced 7 CFU/mL pro-rata 32 bacterial colonies. The broader zone of inhibition created suggests that the chitosan, starch, and kaolin bio-composite can effectively combat bacterial activity.

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