Release property of red ginger essential oil in silica-nanocellulose composite based sachet

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Abstract. Red ginger is an herbal plant that contains active components and can be used to control microorganisms. In this study, active packaging in the form of an antimicrobial sachet was developed. The red ginger essential oil was incorporated into silica-cellulose nanocomposite in powder form in a closed sachet. The silica-nanocellulose composite was fabricated through a sol-gel permeation process in nanocellulose hydrogel. Nanocellulose was isolated from oil palm empty fruit bunches by mechanical treatment, a combination of ultrafine grinding and ultrasonication. Nanocellulose hydrogel was immersed in a sodium silicate solution at three different times. The obtained gel was soaked in 2 M sulfuric acid for 4 hours to produce a silica-nanocellulose composite. Release property of red ginger essential oil from the closed sachet was observed. The effectiveness of the produced antimicrobial sachets depends on the release of the antimicrobial agent from the sachets.

Keywords: Active packaging; Antimicrobial sachet; Nanocomposite; Nanocellulose; Silica

1. Introduction
Food safety is an important requirement that must be attached to food before being consumed by the public. Food packaging materials can play an important role in determining overall food quality and safety. Active packaging involves the interaction between food, packaging materials, and environmental gases from internal packaging. An active packaging system contains active compounds that can affect packaged materials and keep the food free of microorganisms' chemicals and pathogens. The type of antimicrobial packaging can be made by several techniques, including combining antimicrobial agents directly into polymers, coating or adsorbing antimicrobial agents on the polymer surface, using polymers that are naturally antimicrobial, and adding sachets containing volatile antimicrobial agents [1].

Several researchers reported that they successfully produced antimicrobial sachets. Han et al. [2] produced an antimicrobial sachet with microcellular foam starch as matrix and rosemary oil and thyme oil as antimicrobial agents. Seo et al. [3] also reported that a sachet releasing allyl isothiocyanate (AIT)
vapor was developed by encapsulating AIT in calcium alginate beads. Chang et al. [4] produced an antimicrobial sachet with oregano essential oil microencapsulated with polyvinyl alcohol using a spray-drying technique. The release of the active components from the matrix to the packaging's headspace is also affected by the permeability of the sachet material used [5]. The antimicrobial packaging's effectiveness is influenced by many factors, including the antimicrobial agents, the release mechanism, the food chemical properties, the storage, the distribution, and the type of carrier matrix [6].

Silica is a compound produced from the silicic acid polymerization process with a general formula of SiO2. The silica can be found in nature, such as sand, glass, and quartz. Silica is generally widely used as an adsorbent because silica has many advantages: high mechanical strength, good heat resistance, a large surface area, and porous [7].

Porous silica materials have emerged as promising alternatives to polymeric systems due to their unique properties such as high surface area, large pore volume, controllable pore size and structure, versatile functionalization chemistry, excellent biocompatibility and stability, and ease of large-scale preparation at low-cost [8-12]. A few studies have considered using porous silica as a carrier to deliver anti-inflammatory drugs [13,14]. Precipitated SiO2 (silica) has found various applications as filler in rubber and plastics, absorbent, drying powder, a substrate for catalysts, and anti-corrosion agent. Generally, precipitated SiO2 can be produced by mixing aqueous solutions of sodium metasilicate and a mineral acid [15].

Cellulose is a natural polymer that is very abundant in nature, in a linear form consisting of β (1,4)-D-glucan units connected by 1,4-glucoside bonds. Cellulose has very interesting properties such as biocompatibility, biodegradability, and thermal and chemical stability. Cellulose is the main constituent component of green plant cell walls and can also be produced by various living organisms, such as algae, oomycetes, acетobacter, and rhizobium. In general, cellulose content in plants is around 33%, cellulose content in cotton is 90% and wood is 40% -50% [16,17]. The various forms of nanomaterials produced from cellulose are known as cellulose nanomaterials or nanocellulose [18]. The nanocellulose can be isolated from various lignocellulose sources, including the oil palm empty fruit bunches (OPEFBs), which fall into the category of oil palm industry waste. The cellulose content of OPEFBs is about 38.76%, which has a crystalline portion of about 50-90%, and the rest is amorphous. The nanocellulose particles are widely used materials in biotechnology, composites, adsorbents, emulsions and dispersions, and biomedicine because they are easy to form, have a high surface area, and have a high economic value [19]. The nanocellulose can be used as a polymer reinforcing filler because it has high crystallinity, the ratio aspect, the surface area, and the increased biodegradability of the fiber dispersion capability [20]. Nanocellulose, with its nanoscale dimensions, high crystalline nature, and the ability to form hydrogen bonds resulting in a strong network, makes it very hard for the molecules to pass through, suggesting excellent barrier properties associated with films made from these materials [21].

The essential oil shows antimicrobial activity by disrupting the enzyme systems' stability, the bacterial genetic material, and the double layer of phospholipid of the bacterial cell membrane [22]. The red ginger's essential oil consists of α-pinene, camphene, β-pinene, 1.8-cineole, linalool, borneol, neral, nerol, bisabolene, and zingiberene, which can produce the antimicrobial to inhibit the microbial growth [23].

This study's objective was to investigate the release property of red ginger essential oil in a silica-nanocellulose composite based sachet.

2. Materials and methods

2.1. Materials

The materials used for this study were OPEFBs (PTPN VIII, Kertajaya, Lebak, Indonesia) and red ginger essential oil. The chemicals used were sodium hydroxide, H2O2, KCl, sodium silicate 25%, sulfuric acid, and other chemicals for the analysis purposes.
2.2. Preparation of cellulose fibers
OPEFBs were cut into small pieces with a length of 0.5 - 1 cm, then washed with water, then boiled in water (100 ºC) for 1 hour, after that filtered. OPEFBs were then dried in an oven at 55 ºC for 1 day. The 50 g of oil palm fibers were put into 10% sodium hydroxide solution, then heated at 95 ºC for 1 hour. After that, the fibers were washed with deionized water until neutral pH, then dried in an oven at 55 ºC for 1 day.

A two-stage bleaching process was carried out. The first stage of bleaching used H₂O₂ at 95-100 ºC for 1.5 hours, and then the oil palm fibers were filtered and washed with deionized water to pH 4. Then, the second stage used alkaline peroxyde. The alkaline peroxyde solution was obtained by mixing 200 ml of 30% H₂O₂ and 100 ml of 10% NaOH. The cellulose from the first bleaching process was put little by little in the alkaline peroxyde solution and stirred until the cellulose was white. The process was repeated until the fiber color becoming white and then rinsed until the neutral pH.

2.3. Isolation nanocellulose
The nanocellulose was isolated by mechanical treatment using a combination of ultrafine-grinding and ultrasonication [24]. The isolation of nanocellulose was carried out by diluting the cellulose to a concentration of 2% and being suspended using the wearing blender. The isolation was done using the ultrafine grinder with 1500 rpm at the gap level of -5 (15 times), -10 (15 times), -15 (15 times), and -18 (15 times) gradually. Furthermore, the thick suspension was diluted with a ratio of 1:1 (v/v), ultrasonicated with 80% amplitude for 60 minutes to obtain the nanocellulose.

2.4. Preparation of silica-nanocellulose composite as a matrix
A sol-gel permeation process in nanocellulose hydrogels was used to produce silica-nanocellulose composites. The nanocellulose hydrogel was prepared using a vacuum filter for 7 hours. Then, the resulting hydrogel was immersed in the sodium silicate solution for 2, 4, and 6 hours. The obtained gel was drained and dried for 15 hours at room temperature. Subsequently, the gel was immersed in a 2 M sulfuric acid solution for 4 hours to produce a silica-nanocellulose composite. The nanocomposite produced was washed to remove the residual sulfuric acid and the generated sodium sulfate.

2.5. Production of antimicrobial sachet
The red ginger essential oil was added into silica-nanocellulose composite powder (80 mesh). Then, one gram of silica-nanocellulose composite powder contained red ginger essential oil was put into the LDPE sachet with a size of 3 x 3 cm and sealed.

2.6. Characterization
The morphology of the obtained nanocellulose and nanocomposites was analyzed by scanning electron microscope (SEM, SEM Zeiss EVOMA10, Cambridge, England) at 16 kV. The size of the nanocellulose was measured using the Image J software. The spectra of FTIR of cellulose, nanocellulose, silica, and its nanocomposites were obtained using Bruker Tensor 37 (Billerica, MA, USA) in absorbance mode with a resolution of 4 cm⁻¹. GC-MS analyzed the produced sachets to know what compounds were released from the antimicrobial sachets.

2.7. Release of the antimicrobial agent from antimicrobial sachet [14]
The release of the antimicrobial agent from the antimicrobial sachet was evaluated by conditioning at RH values of 84% (using dissolved KCl in 400 ml distilled water until saturated in a desiccator). The produced sachet was put into the desiccator. Then, the sachet was weighed every day for 7 days to determine the changes in sachet weight.
2.8. Maximum adsorption
A total of 200 mg of the silica-nanocellulose composite powder were put into the test tube. The red ginger essential oil was then added 1 ml into it and allowed to soak for 5 minutes. Subsequently, the solids and the liquid were separated using a 100-mesh filter. Then, the solids obtained were weighed.

2.9. Test of Gas Chromatography-Mass Spectrometry (GC-MS) [25]
The testing using GC-MS was carried out on the three sachet samples, namely A, B, and C, put into an airtight bottle. Sample A is a sachet containing only red ginger essential oil as an antimicrobial agent without a matrix, while samples B and C are sachets containing an antimicrobial agent incorporated in a silica matrix and a silica-nanocellulose composite matrix, respectively.

3. Results and discussions
Fig. 1 shows an SEM image of nanocellulose isolated from OPEFBs. In this study, nanocellulose used to fabricate silica-cellulose nanocomposite had a diameter of about 46.32±17.43 nm with a length of several micrometers (Fig. 1). The size reduction using the ultrafine grinder was made by passing the cellulose suspension between static and rotating grindstones several times to break down the cell wall structure of lignocellulosic materials [26]. Nanocellulose produced from the ultrafine grinding process is still not a single fiber with a size that is not homogeneous, so ultrasonication is needed. Because of the cavitation effect in nanocellulose suspension during the ultrasonication process, the formation of bubbles produces high ultrasonic energy and makes the particles more homogeneous [27].

![Figure 1. SEM image of nanocellulose from OPEFBs.](image)

Fig. 2 shows the FTIR spectra of cellulose, nanocellulose, silica, and its nanocomposite. Cellulose and nanocellulose have identical FTIR profiles. In FTIR spectra of cellulose, there was no peak at around 1267, 1509, and 1720 cm\(^{-1}\) indicating C–O stretching of hemicelluloses and lignin, C=C aromatic skeletal vibration of lignin, and C=O stretching of hemicelluloses and lignin, respectively [28]. This means that the alkaline and bleaching treatment removed lignin and hemicellulose completely.
Figure 2. FTIR profiles of cellulose, nanocellulose, silica, and its nanocomposite

The antimicrobial sachet matrix was obtained by immersing the nanocellulose hydrogel in sodium silica silicate solution for 2, 4, and 6 hours. During the soaking, the sodium silicate solution diffused into the hydrogel due to the difference in concentration so that there was a material exchange between water and sodium silicate solution in the hydrogel. The hydrogel was then dried and immersed in a 2 M sulfuric acid solution for 4 hours. During the immersion, there was a reaction between the sodium silicate and the sulfuric acid, which can be seen in equation one, which produces the water [29]. Then, the silica-nanocellulose composite was rinsed with the distilled water to remove the formed Na₂SO₄ (sodium sulfate), leaving the silica-nanocellulose composite.

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\text{Na}_2\text{SiO}_3 + \text{H}_2\text{SO}_4 = \text{SiO}_2 + \text{Na}_2\text{SO}_4 + \text{H}_2\text{O} \text{ (air)}
\]  

(1)

The dried hydrogel and produced silica-nanocellulose matrix can be seen in Fig. 3. The silica-nanocellulose composites with different immersion times in sodium silicate solution were fragile and white.

Figure 3. Dried-hydrogel (a), hydrogel immersed in sodium silicate for 2 h (b), 4 h (c), 6 h (d).

It seemed that with a longer immersion time of the hydrogel in sodium silica solution, the more silica entered into the nanocellulose hydrogel. Therefore, the percentage of silica in hydrogel became higher. With 2 hours’ immersion time, the hydrogel had the lowest silica percentage (76.72%) and followed by the immersion of 4 hours and 6 hours with 84.83% and 86.35%, respectively.

Fig. 4 shows SEM images of the silica-nanocellulose composite matrix produced by immersing in the sodium silicate solution for 2, 4, and 6 hours. All matrices were porous. The silica-nanocellulose
composite matrix before the addition of the essential oil can be seen in Fig. 4a, 4c, and 4e. However, after the addition of the essential oil, the matrices seem to expand, which are shown in Fig. 4b, 4d, and 4f. The swelling in the matrix was caused by the essential oil entering the matrix pores.

**Figure 4.** SEM images of the silica-nanocellulose composite matrix produced by immersing in the sodium silicate solution for 2 h (a, b), 4 h (c, d), and 6 h (e, f): without essential oil (left) and with essential oil (right)

The absorption test aims to find out how much oil was absorbed by the matrix. The nanocomposite matrix immersed in sodium silicate solution 2 h was able to absorb the oil as much as 56.53% from the initial weight, followed by the matrix immersed 4 h, which was able to absorb as much as 82.24%, and the matrix immersed 6 h as much as 94.38%. From these data, it can be concluded that the longer immersion time in the sodium silicate solution, the matrix structure will be more porous. Therefore, more oil will be absorbed by the matrix. The addition of the essential oil change not only the weight but also the color of the matrix; this can be shown in Fig. 5. Before adding the essential oil, the matrix was white and dry, but it became yellow after the addition of the essential oil. This indicated that the essential oil added was trapped by the silica-nanocellulose composite matrix.
Figure 5. antimicrobial sachet without essential oil (A), with essential oil (B)

A release test was carried out to determine the release of essential oil from the composite matrix. The release property of the composite matrix is time-dependent. Seo et al. [30] reported that the characteristics of the release of volatile compounds from antimicrobial sachets are influenced by incubation time, RH, and temperature.

Fig. 6 shows the release of red ginger essential oil from antimicrobial sachets at 85% RH and 30 °C using a matrix (silica and silica-nanocellulose composites) and a sachet without using a matrix. Antimicrobial sachets without using a matrix could release higher essential oils than those of the antimicrobial sachets using the matrix. This was because the essential oils in sachets without a matrix were not blocked by any volatile oil. Meanwhile, antimicrobial sachets containing the essential oil incorporated in the composite matrix were more difficult to evaporate because the nanocellulose inhibited them. In addition, the type of matrix also influenced the release of red ginger essential oil in sachets. The red ginger essential oil in antimicrobial sachets using silica-nanocellulose composite matrices was more difficult to evaporate than sachets using silica matrices. This is because the addition of nanocellulose could inhibit the rate of release of essential oils. After all, nanocellulose had a function as a barrier in the matrix. An illustration of the mechanism for releasing essential oils for each type of sachet can be seen in Fig. 7.

Based on the observations shown by Fig. 6, it can be seen that there was an increase in the concentration of essential oils released by sachets from day to day. This shows that a component had disappeared from the sachet. The missing component was the essential oil added to the matrix. Sachets with silica-nanocellulose composite matrix released less oil than sachets without matrix and silica matrix. This was because essential oil in sachets without a matrix was more volatile, and the essential oil directly diffused out from the sachet. Meanwhile, in a sachet with a matrix, the essential oil was released, leaving the matrix, and then to the sachet's headspace and then released from the sachet.
Figure 6. The release of red ginger essential oil from antimicrobial sachets at 85% RH; 30 °C using a matrix (silica, silica-nanocellulose composites) and a sachet without using a matrix.

Figure 7. The release mechanism of antimicrobial sachets without matrix (A); antimicrobial sachets with silica (B) matrix and; antimicrobial sachets with silica-nanocellulose (C) composite matrix.

GC-MS testing was carried out to find out what compounds evaporated from antimicrobial sachets. A total of 26 types of compounds were detected from the antimicrobial sachets without matrix (sachet A). Meanwhile, sachets using silica matrix released 18 compounds, less than the volatile oil contained in original essential oils (Table 1. This indicated that the matrix used could inhibit the release of some compounds contained in essential oil. In addition, antimicrobial sachets using silica-nanocellulose composite matrix released the lowest compounds, only 16 types of compounds. Monoterpenes and sesquiterpenes were the main components of essential oils. The results of GC-MS tests detected mostly monoterpenic compounds, which have a C-atom number of 10-14. Meanwhile, the detected sesquiterpenes are Borneol, β-bisabolene, and β-Sesquiphellandrene with a large number of C atoms 15. Other cyclic compounds like zingiberene are not detected because the matrix still traps them in sachets.

The black color in Table 1 is a component of essential oil compounds that were not detected when GC-MS testing was performed.
Table 1. The compounds of each GC-MS test sample.

| Compounds                  | Sachet A | Sachet B | Sachet C |
|----------------------------|----------|----------|----------|
|                            | RT       | Area (%) | RT       | Area (%) | RT       | Area (%) |
| **Acetone**                | 1.54     | 3.93     | 1.53     | 1.71     | 1.53     | 6.00     |
|                            | nd       | nd       | nd       | nd       | 1.56     | 1.35     |
|                            | 1.69     | 0.16     | 1.68     | 0.31     | nd       | nd       |
|                            | nd       | nd       | 1.72     | 0.21     | nd       | nd       |
| **Hexane**                 | 1.78     | 0.18     | 1.77     | 0.25     | nd       | nd       |
| 2-methyl-3-buten-ol        | 1.83     | 1.00     | 1.82     | 0.49     | 1.82     | 0.45     |
| methlycyclopentane         | 1.91     | 0.13     | 1.90     | 0.31     | 1.90     | 0.25     |
| **Benzene**                | 2.92     | 0.68     | 2.91     | 0.60     | 2.91     | 2.36     |
| **Hexanal**                | 3.27     | 0.28     | nd       | nd       | nd       | nd       |
| 2-heptanone                | 4.72     | 0.36     | 4.71     | 0.33     | nd       | nd       |
| **Tricylene**              | 5.42     | 1.64     | 5.41     | 1.64     | 5.41     | 1.56     |
| α-pinene                   | 5.67     | 16.34    | 5.66     | 16.60    | 5.66     | 14.16    |
| **Camphene**               | 6.01     | 59.28    | 6.00     | 61.43    | 6.00     | 58.11    |
| 6-methyl-2-heptanone       | 6.09     | 0.21     | nd       | nd       | nd       | nd       |
| B-pinene                   | 6.66     | 0.99     | 6.66     | 0.98     | 6.65     | 0.42     |
| 6-Methyl-5-hepten-2-one    | 6.85     | 2.75     | 6.84     | 2.84     | 6.84     | 2.34     |
| B-myrcene                  | 6.96     | 1.80     | 6.95     | 1.82     | 6.95     | 1.88     |
| o-cymene                   | 7.84     | 0.27     | nd       | nd       | 7.83     | 0.48     |
| 2-bornene                  | 7.95     | 0.27     | 7.94     | 3.23     | 7.94     | 2.87     |
| 1,3,3-Trimethyl-2-oxabicyclo[2.2.2]octane | 8.01 | 4.94 | 8.00 | 5.34 | 8.00 | 5.12 |
| **Borneol**                | 11.72    | 0.30     | nd       | nd       | nd       | nd       |
|                            | nd       | nd       | 19.64    | 1.50     | 19.64    | 2.14     |
| β-bisabolene               | 20.16    | 1.07     | 20.05    | 0.41     | 20.04    | 0.48     |
| β-Sesquiphellandrene       | 21.00    | 0.21     | nd       | nd       | nd       | nd       |

4. Conclusion
The silica-nanocellulose composite matrix was successfully fabricated by immersing the nanocellulose hydrogel in the sodium silicate solution. The immersion of the hydrogel in the sodium silica solution affected the absorption ability of the produced matrix. The hydrogel immersed for 2 hours was able to absorb the essential oil of 56.53%, while the soaking of 4 and 6 hours were able to absorb 82.24% and 94.37%, respectively.

5. Conflicts of interest
The authors declare that there is no conflict of interest regarding the publication of this manuscript.
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