Journal of Fibers and Polymer Composites 3 (1): 62-73 (2024)

Journal of Fibers and Polymer Composites

https://journals.gesociety.org/index.php/jfpc/index

THE CHEMICAL AND WATER SORPTION PROPERTIES OF CHEMICALLY MODIFIED *Sesbania grandiflora* AND *Leucaena leucocephala* FIBERS AND THEIR OPPORTUNITIES AS BIOCOMPOSITE FILLERS

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Abstract. *Researchers are interested in investigating the optimum features of Sesbania grandiflora fibers (SGs) and Leucaena leucocephala fibers (LLs) for application in a wide range of applications. The purpose of this study was to explore the effects of NaOH and bleaching on the chemical characteristics, density, moisture content, and functional groups of sesbania g. and leucaena l. fibers. SGs and LLs fibers were chemically changed in various ways, including alkalization with 5% NaOH for 2 hours and bleaching with sodium chlorite ($NaClO_2$) and acetic acid (CH_3COOH). The results of the study showed that the cellulose, lignin, and hemicellulose content of the modified fiber was generally lower than that of the raw fiber. This was explained by the hydrolytic agents NaOH and bleaching causing damage to the cellulose chains and bond modifications. It was discovered that NaOH treatment was more effective at removing lignin and hemicellulose components in addition to cellulose, based on the functional groups seen in FTIR spectra. After the alkali and bleaching processes, the water content of SGs/NaOH, LLs/NaOH, SGs/bleaching, and LLs/bleaching fibers was reduced to 5.4 ± 0.89 %, 6.23 ± 0.87 %, 7.12 ± 1.02 %, dan 0.22 ± 0.21 %, respectively. This is also because the fiber has a relatively high fiber density of between 0.2 and 0.3 g/cm3, which is caused by the loss of non-fiber substances that are linked to the fiber. The results show that PFs treated with bleaching, NaOH, and raw fibers have the potential to be used as fillers in biocomposites and as substitute materials for glass fiber in the textile industry.*

Keywords: *Leucaena leucocephala fibers; Sesbania grandiflora fibers; chemical properties; density; moisture content, FTIR.*

1. Introduction

Natural fibers are still the focus of research by academics and scientists for industrial applications because they have a unique structure, are cheap, abundant, non-toxic, and eco-friendly, such as corn husk fiber, *Paederia foetida* fiber, banana stem fiber, pandanwangi fiber, and so on [1]-[4]. Every part of the plant is known to be a source of fiber, such as tree bark, leaves, and seeds/fruit, and this has opened up prospects for additional natural fibers to be researched for their fibers, such as *Leucaena leucocephala* and *Sesbania grandiflora* fibers.

Received January 4, 2024; Accepted March 6, 2024; Published March 29, 2024
https://doi.org/10.55043/jfpc.v3i1.146

L. leucocephala is a shrub in the Leguminosae family that may grow and thrive in Indonesia. This plant grows swiftly, reaching a height of 8-15 m and having many branches. *L. leucocephala* is frequently considered a weed due to its ease of reproduction. Even if this plant is clipped, cut down, or burned, its shoots will regrow in vast numbers. It has been utilized as a shade tree, green manure, roadside plant, firewood, and a source of feed for cattle [5], [6]. Unfortunately, this ubiquitous and environmentally friendly substance is still underutilized. According to Suraj *et al*. [5] the seeds of *L. leucocephala* contain 25.9% amino acids with a composition of 1.7% lysine, 0.7% histidine, 2.2% aginine, 3.6% asparcate, 0.7% threonine, etc., and have the potential as a raw material for meat analogues. Shekhar *et al*. [6] retrieved the 4CL enzyme as the target gene from *Leucaena leucocephala* trees to generate pulp raw materials with good felting strength, Runkel ratio, tensile strength, and so on. However, prior studies reveal that there is a dearth of information and research concerning the chemical and physical characteristics of the fibers found in the stems of *L. leucocephala* plants.

S. grandiflora is a type of legume plant that is extensively recognized in Indonesia, particularly in central and eastern Indonesia. *S. grandiflora*. contains important phytochemicals such as alkaloids, terpenoids, flavonoids, steroids, anthraquinones, catechins, phytosterol tannins, and so on [7]. We discovered several prior scholars who had researched the chemical qualities of *S. grandiflora* nuts, and it was stated that *S. grandiflora* nuts had 10.14% water, 7.10% fat, 36.21% protein, and 12.64% crude fiber. Ishartani *et al*. [8] studied the influence of boiling and soaking treatments on the chemical qualities (ash, fat, protein, phytic acid, and cyanide acid) and physical properties (degree of whiteness, kamba density, and solubility) of white flowering turmeric bean flour. They found that while the flour had the same kamba density after soaking for 24 hours and boiling for 90 minutes, it had less ash, fat, protein, phytic acid, HCN, and whiteness. The nickel oxide nanoparticles (NiO NP) found in *S. grandiflora* flowers were investigated by Gobinath *et al*. [7]. NiO NPs have a rod-like form and their crystallite size was found to be between 19.9 and 60.4 nm. Several searches revealed that there is no information about the features of *S. grandiflora* stem branches, despite the fact that *S. grandiflora* stem branches have the ability to generate abundant natural fiber.

Even though natural fibers have numerous advantages with their greatest features, they are less chosen due to their hydrophilic properties, and their use is frequently limited to interior structures [9], [10]. As a result, natural fibers require treatments to generate a variety of qualities suited for a variety of end purposes. Fortunately, these qualities can be enhanced by using chemical treatments such as NaOH and bleaching. Natural fibers are routinely treated with NaOH in the textile and materials industries to get desired qualities. Meanwhile, the bleaching process with acetic acid normally seeks to whiten and clean natural fabrics. Sari *et al*. 2023 [2] observed that

after treating *Paederia foetida* fibers (PFs) with NaOH and KOH, the tensile strength of the fibers rose by approximately 43.27%, with the greatest crystallinity index achieved from *P. foetida* fibers treated with KOH at 79.7%.

Then, Syafri *et al*. [11] produced nanocellulose from *Agave gigantea* (AG) fibers using mercerization and bleaching methods to produce cellulose micro fiber (CMF), and they reported that the CMF crystal index is known to reach 48.3%, with a cellulose content of 20.4%, making it suitable for use as a reinforcement in polymer biocomposites. Syafri *et al*. 2023 [12] employed *Agave Gigantia* cellulose micro fiber (CMF) with a fiber diameter of 10-15 m to reinforce poly vinyl Acetate (PVA) composites. In order to reinforce poly vinyl Acetate (PVA) composites, Agave Gigantia cellulose micro fiber (CMF) with a fiber diameter of 10-15 m was used by Syafri *et al*. 2023 [12]. Tensile strength of 10 g of PVA was found to be 43% higher in the PVA/U2 AG biocomposite film than in the untreated PVA/CMF film. This was achieved by mixing 10 g of PVA. Previous research has shown that surface modification of natural fibers using alkalization and bleaching methods is applicable to other natural fibers where structural changes caused by chemical treatment can modify the chemical and physical properties of natural fibers.

Consequently, this work aims to give a comprehensive understanding of the chemical and physical characteristics of fibers from *S. grandiflora* and *L. leucocephala*. To change the surface of the two distinct fibers, alkalization with 5% NaOH and bleaching with $NaClO_2$ and acetic acid (CH_3COOH) were used. The chemical characteristics, functional groups, densities, and moisture content of two different types of fibers were investigated. The results obtained from this investigation may contribute to the development of a new filler composite.

2. Materials and Methods

2.1. Materials

L. leucocephala (Figure 1a) and *S. grandiflora* (Figure 1b) stem branches were collected from Lombok, West Nusa Tenggara, Indonesia. Acetic acid (CH_3COOH), sodium hydroxide (NaOH), sodium chlorite ($NaClO_2$), and analytical grade distilled water with a 99.8% weight fraction purity were supplied by PT. Indo Acidatama Tbk, Jakarta, Indonesia.

2.2 Extraction of fibers

The dried stem branches of *S. grandiflora* and *L. leucocephala* were prepared separately and cleaned of dirt. They were cut into small pieces using a knife with a size of 1 cm, then crushed using a crushing machine to get more uniform small fibers. The raw S. grandiflora and raw L. leucocephala fibers are mechanically processed, then filtered through a 20-mesh sieve, dried in an oven at 105 ^oC for 60 minutes, and stored in an airtight plastic container to prevent moisture damage.

Figure 1. (a) *Leucaena Leucocephala*, and (b) *Sesbania Grandiflora* fibers

2.3 Alkali and bleaching treatment of fibers

Different SGs raw and LLs raw were alkalized and delignified in this technique. To begin the alkalization or mercerization process, immerse 200 grams of SGs and 200 grams of LLs in a 5% NaOH solution in a separate container for 120 minutes. After that, distilled water was used to filter and wash the SGs and LLs five times in order to bring their pH down to 7. By soaking them in a $NaClO_2$:CH_3COOH (4:1) solution, they were bleached (Figure 2) [11], [13]. Before being stored in an airtight container, they were additionally dried in an oven set to 60 °C for six hours.

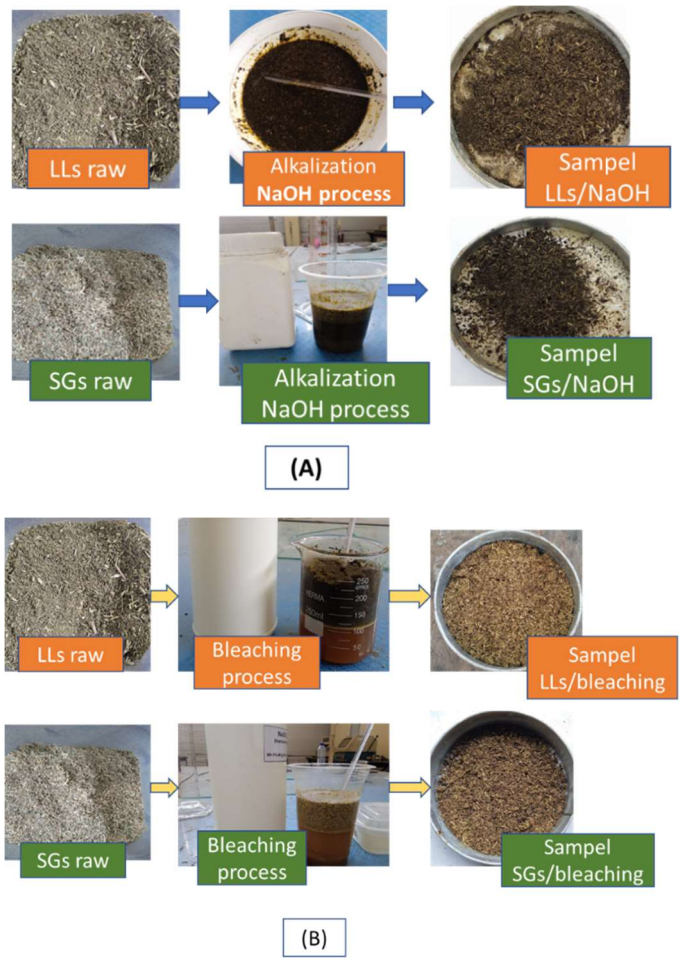

Figure 2. *Sesbania grandiflora* and *leucaena leucocephala* fibers, (a) alkalization, and (b). Bleaching process

2.4 Characterization

2.4.1 Fourier Transform Infrared (FTIR) spectroscopy

The FTIR spectra were used to identify whether the samples contained free functional groups. Thermo Scientific Smart iTR devices with horizontal attenuated total reflectance (ATR) were used to generate FTIR spectra. The ATR crystal was used to mount the samples, and transmittance mode was used to gather the spectra. An average of 32 photos at a resolution of 4 cm-1 was used to create each spectrum [14]. The samples were crushed with a crusher and pestle into incredibly fine particles.

2.4.2 Chemical content

The chemical compositions of SGs raw, SGs/NaOH, and SGs/bleaching, as well as LLs raw, LLs/NaOH, and LLs/bleaching, were measured experimentally. The contents of cellulose and lignin were determined using the Association of the Pulp and Paper Industry (TAPPI) T 9M-54 and TAPPI T 13M-54 standards, respectively. American Standard Testing Materials (ASTM) 1104-56 is used to quantify hemicellulose.

2.4.3 Density

Before doing a density test, each sample was oven-dried for 12 hours at 105 °C to remove any water that the samples may have absorbed. As stated by Sari *et al*. [4], each sample was assessed using a liquid immersion test with a pycnometer and methanol. To find each sample's average density, the four were first weighed using an analytical balance with a resolution of 0.001 mg.

2.4.4 Moisture Content

Every sample—raw fiber and treated fiber—was boiled to 60 °C for five hours in an oven. The sample's weight was recorded both before (M_a) and after (M_s) it was placed in the oven. Equation (1) was then used to calculate each sample's water content: [15].

$$\text{Moisture content (\%)} = (M_s - M_a)/ M_s \times 100 \quad (1)$$

3. Results and Discussion

3.1 FTIR analysis

Figure 3 displays the LLs' FTIR spectra both before and after alkali treatment and bleaching. The patterns are the same in all of the examples. Hydrogen bonding from the hydroxyl group and cellulose I/O-H stretching vibrations are what generate the broad band in the 3000-3500 cm^{-1} region. Raw SGs (Figure 3) and raw LLs (Figure 4) show a band in the 1713 cm^{-1} region, but this band disappears after alkali treatment and bleaching, which is connected to the C=O stretch of hemicellulose [16], [17]. Table 1 summarizes the assignment peak positions of raw SGs,

SGs/NaOH, and SGs/bleaching fibers. Meanwhile, Table 2 shows the entire spectra of LLs samples before and after NaOH and bleaching treatment.

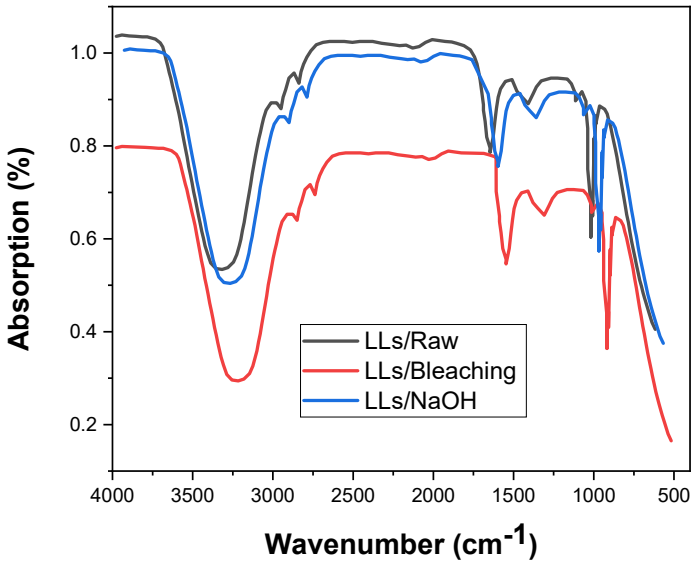

Figure 3. FTIR of *L. leucocephala* fibers with different treatments.

Table 1. Peak positions and chemical group assignments of LLs raw, LLs/NaOH, and LLS/bleaching.

Wave number (cm^{-1})			Assignments
LLs Raw	**LLs/NaOH**	**LLs/ bleaching**	
3319.56	3275.24	3204.65	hydrogen bonding of hydroxyl groups and cellulose Iβ/O-H stretching vibrations of α-cellulose.
2950.21	2895.21	2845.14	The C-H stretching vibrations seen in cellulose and hemicellulose components.
1755.13	-	-	CO stretching vibration of carboxylic acid and hemicellulose ester groups is known as hemicellulose/CO stretching.
1526.26	1585.23	1545.01	CO stretching of lignin
1405.47	1350.48	1310.26	Bending of aromatic lignin/CO
1105.06	1059.92	1009.85	The C-O stretching vibrations found in cellulose, hemicellulose, and lignin.
1020.52	960.6	915.46	Symmetrical CO stretching in lignin

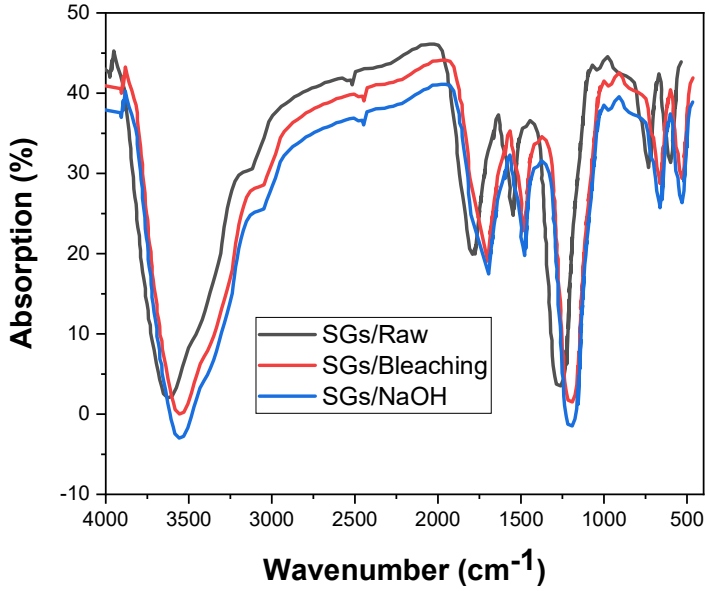

Figure 4. FTIR of *S. grandiflora* fibers (SGs) with different treatments.

Table 2. Peak positions and chemical group assignments of raw SGs, SGs/NaOH, and SGs/bleaching.

Wave number (cm^{-1})			Assignments
SGs Raw	**SGs/NaOH**	**SGs/Bleaching**	
3619.97	3550.21	3550.21	Hydrogen from hydroxyl groups and O-H stretching vibration of α-cellulose bonds.
-	3044.59	3044.59	components of hemicellulose and cellulose as well as the C-H stretching vibrations found in them.
1779.75	-	-	stretching vibrations of the ester groups in hemicellulose and carboxylic acid CO in hemicellulose/CO.
-	1695.21	1695.21	Absorbed water
1545.01	-	-	Lignin and aromatic Lignin exhibits C=C stretching.
-	1475.24	1475.24	Cellulose/CH_2 bending
1270.04	1185.49	1190.42	Lignin/-CO stretches from the acetyl group
1030.37	965.53	960.6	Stretching vibrations in cellulose (C-O), hemicellulose, and lignin.

3.2 Chemical properties

Table 3 demonstrates that the cellulose content of the SGs/NaOH and LLs/NaOH samples is lower after NaOH treatment than that of the raw SGs and raw LLs. The NaOH solution acts as a hydrolytic agent, physically or chemically damaging the cellulose chains and changing the linkages. It is assumed that its presence in the form of a strong alkali solution causes polysaccharide bond hydrolysis in fibers such as cellulose, hemicellulose, and lignin. Apart from that, it is believed that the 5% NaOH solution dissolved the non-cellulose components of the fiber, resulting in a decrease in the cellulose, lignin, and hemicellulose content of the fiber. Sari *et al*. [4] reported that treatment with a high concentration of NaOH can damage the cell wall because of excessive extraction between lignin and hemicellulose. NaOH (sodium hydroxide) acts as a bond-breaking or bond-removing agent between cellulose polymer chains when natural fibers are treated with it, particularly at high temperatures and concentrations. As a result, the glycosidic connections tying the glucose units in the cellulose structure dissolve, which ultimately leads to the cellulose fibers' lengthy structure disintegrating or being destroyed.

Despite having less cellulose than before, the resulting natural fibers have a variety of chemical and physical characteristics, lessen their negative effects on the environment, and produce less waste. Applications like packaging and textiles, where end-of-life disposal and sustainability are crucial, greatly benefit from these attributes.

Table 3. Chemical composition of *S. grandiflora* and *L. leucocephala* fibers with different chemical treatments.

Codes Samples	Hemicellulose (%)	cellulose (%)	Lignin (%)
LLs Raw	9.85	20.24	8.71
LLs/NaOH	2.41	6.65	0.91
LLs/Bleaching	2.33	7.39	0.31
SGs Raw	12.58	26.13	7.79
SGs/NaOH	2.3	8.14	1.12
SGs/Bleaching	2.28	8.84	1.59

Furthermore, the cellulose, lignin, and hemicellulose levels of the LLs/bleaching and SGs/bleaching samples were found to be the lowest of the samples investigated (see Table 3). The oxidation process between lignin and acetic acid is thought to be the source of this decline in lignin concentration. This process produces molecules that can dissolve in water and be liberated from the fiber. In some circumstances, such as in the textile industry, lignin must be eliminated in order to generate whiter fibers or expose them to better dyeing. Acetic acid is utilized to oxidize and

eliminate lignin from natural fibers during this bleaching procedure [18]. Furthermore, hemicellulose dissolves in acetic acid and is eliminated from natural fibers, resulting in low hemicellulose content of 2.28% and 2.33% in LLs/bleached and SGs/bleached fiber samples, respectively. The process of bleaching with acetic acid causes chemical structural changes in cellulose, which is the main ingredient in fiber. Acetic acid alters both the chemical and physical properties of cellulose by affecting intramolecular linkages. As a result, cellulose becomes more easily oxidized, resulting in a low cellulose content of roughly 8.84% and 7.39%, respectively. Although the bleaching process can reduce chemical contents such as cellulose, lignin, and hemicellulose, this decrease is frequently sought in certain industries, such as the textile industry, to attain desirable fiber qualities.

3.3 Density and moisture content analysis

Table 4 demonstrates that following NaOH treatment and bleaching, the density of the SGs/NaOH and LLs/NaOH samples is higher than that of the raw LLs and raw SGs samples. Increasing density values in SGs/NaOH and LLs/NaOH samples can arise for a variety of reasons, including: (1). Fiber swelling: When natural fibers are soaked in NaOH solution, sodium hydroxide reacts with fiber components such as cellulose and hemicellulose, causing natural fibers to swell. This occurs because NaOH can dissolve hydrogen bonds in the fiber structure, causing changes in molecular order and an increase in fiber volume. As a result, the density of natural fibers increases after NaOH treatment. (2). Water absorption: NaOH is a hygroscopic substance, which means it may absorb water from the surrounding air. When the fibers are treated with NaOH solution, they can absorb water from their surroundings, resulting in an increase in moisture content. This can result in an increase in the weight of natural fibers and, as a result, an increase in density. Meanwhile, the increase in density value of the LLs/bleaching and SGs/bleaching samples is assumed to be related to fiber swelling. Acetic acid therapy causes edema in both LLs and SGs. Acetic acid is a water-soluble chemical that absorbs moisture. Acetic acid's capacity to absorb water can cause fibers to swell when exposed to it. This swelling is hypothesized to play a role in the increased density of LLs and SGs fibers. The density of the fibers examined was found to be lower when compared to glass fiber density of 2.54 g/cm^3 [19] and corn husk fiber treated with 8% NaOH density of 0.61 g/cm^3 [4].

Table 4 also demonstrates that the moisture content value of the fiber after NaOH and bleaching treatment (see LLs/NaOH samples, LLs/bleaching, SGs/NaOH and SGs/bleaching) is lower than LLs raw and SGs raw. This is assumed to happen because most of the non-fiber (non-cellulose) components, such as hemicellulose, dissolve in NaOH and bleaching chemicals, causing the fiber's water content to drop.

Table 4. The moisture content and density of fibers from *L. leucocephala* and *S. grandiflora*

Codes Sample	Moisture content (%)	Density (g/cm^3)
LLs Raw	7.7 ± 0.34	0.25 ± 0.32
LLs/Bleaching	6.4 ± 0.9	0.3 ± 0.11
LLs/NaOH	6.23 ± 0.87	0.28 ± 0.21
SGs Raw	9.8 ± 0.78	0.2 ± 0.13
SGs/Bleaching	7.12 ± 1.02	0.22 ± 0.21
SGs/NaOH	5.4 ± 0.89	0.26 ± 0.5

4. Conclusions

We examined the chemical characteristics, density, functional groups, and moisture content of *Leucaena leucocephala* (LLs) and *Sesbania grandiflora* (SGs) fibers treated with NaOH and bleaching. After being treated with NaOH and bleaching, the cellulose, lignin and hemicellulose content and water content became low. The higher density value were obtained from samples LLs/NaOH and LLs/bleaching reached 0.2 - 0.3 g/cm^3, compared to raw LLs; which is attributed to the loss of non-fiber substances bound to the fiber and the drying of water remaining in the natural fiber after the NaOH and bleaching processes. In some circumstances, a low-density value is desired to suit specific requirements in businesses that use natural fibers, such as textiles or paper manufacture.

5. Declaration of competing interest

The authors declare that they have no known competing interests that could have influenced the literature reported in this paper.

6. CRediT authorship contribution

Sari, N.H.: Conceptualization, Writing – original draft, review & editing, Supervision, Funding acquisition. Suteja, S and Sutaryono, Y.A: Resources, Investigation. Data curation, Visualization.

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