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Identification and characterization of mineral content in the white sand of the beach of Talaud Island

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Abstract
The Talaud Archipelago is one of the Regions in North Sulawesi with natural materials such as white sand which is very potential. The purpose of this study was to identify the mineral content and to determine the characteristics of silica extracted using the alkaline fusion method on the white sands of Sara Beach and the White Sands of the Talaud Islands. The analysis used is FTIR (Fourier Transform Infra Red). The results of the FTIR analysis show that the spectrum results that often appear in the wave number 680-850 cm-1 which is indicated as a symmetric stretching vibration function group of Si-O in siloxane (=Si-O-Si=), wave number 1800-1600 cm-1 which is the –OH bending vibrational functional group of Silanol (Si-OH).

Keywords: white sand, mineral content, FTIR

Introduction

Talaud Islands Regency is an area that has a wide distribution of white sand with a coastline length of 367.7 km (Central Bureau of Statistics of the Talaud Islands Regency, 2018). The existence of sand that is widely distributed and its abundance is an attraction. However, these natural resources have not been used optimally and are considered not to have high economic value. In fact, if an exploration of the mineral content contained in white sand is carried out, the local community will use white sand as an economical material. For this reason, it is necessary to study its utilization in order to increase the added value of white sand. One of the minerals contained in white sand is silica (SiO2) which has very potential value.

In recent years, the use of silica sand as a raw material for ceramic composites, especially as a raw material for making fuel cell seals, has been widely carried out and is an interesting study and provides added value for silica sand (Dewa and Pasaribu, 2020) [2]. On a larger scale, refined silica material is very useful to meet increasingly complex human needs. In the energy sector, the use of silicon is the raw material for the manufacture of solar panels that require a high level of purity (Rifina *et al*, 2017).

In nature, silica is difficult to obtain as a high-purity element, because it has a high affinity for oxides and other atoms with high electronegativity. Chemically, the bond between oxygen and silicon is 50% covalent and 50% ionic, thus forming a strong bond (White, 2005). The impurities contained in silica sand can affect the quality of silica sand and products made from silica sand such as damaging the transmission of optical fiber and transparency in the glass industry, blackening ceramic products and lowering the melting point of refractory materials. So that in its use, silica sand needs to be purified first (Januarty and Yuniarty, 2015). Silica purification is usually done by adding hydrochloric acid (HCl). This purification produces silica with a content of 99.99% (Sulistiyono *et al*, 2000).

In addition, one way to obtain silica from a natural material is to use the extraction method. This method is used to separate solutes between two immiscible liquids. The conditions of the extraction process which include temperature, solvent concentration, extraction time, and stirring are factors that greatly affect the success of the extraction process (Adziima *et al*, 2013). While the principle of the alkali fusion method is to dismantle the chemical bonds in the material by using alkaline compounds such as KOH, NaOH, Na2CO3 and then bind to silica (Fauziyah, N.A, 2015)

So far, testing or identification of mineral content or extraction of silica content has not been applied to the white sands of the Talaud Islands. Therefore, based on the presence of natural materials, in this case the abundance of white sand in the Talaud Islands Regency, the author intends to conduct research on the identification and characterization of the silica (SiO2) content in the white sands of the Talaud Islands.

Research and Methods

The sample in this study was white sand which was taken at five different points along the coast of Sara Island, Lirung District and at Pair Putih beach, South Bitunuris Village, Salibabu District, Talaud Islands Regency.

Tools and Materials

Tool: Balance, Filter paper, Beaker, Pyrex brand, Measuring Cups, Erlenmeyer flask, and Approx brand, Mortar and pestle, Funnel, Stirring rod, Sieve 40 mesh, Thermometer, Universal indicator, set of titration tools, instruments: FTIR, XRF, and XRD.

Material

White sand as a source of silica (SiO2), HCl (hydrogen chloride), NaOH, aquades

Data Collection Techniques

Data collection is based on work procedures carried out as follows.

The same working procedure was carried out on 2 types of sand originating from two different places, namely Sara Island beach sand, Lirung District and White Sand beach sand, South Bitunuris Village, Salibabu District, Talaud Islands Regency.

1. Sand Preparation

A number of white sand is cleaned of impurities and crushed with a mortar and pestle then sieved through a 40 mesh sieve. The sieve results identified mineral content using XRF.

The white sand is first washed with distilled water and then cleaned/separated from impurities (Wianto *et al*, 2010). White sand is ground using a mortar and pestle, then sieved with a 40 mesh sieve (Langi *et al*, 2020) [4].

2. Identification of Sand Minerals

A total of 50 grams of sieved quartz sand powder was soaked with 100 mL HCl 2 and 3 M in a beaker glass for 14 hours to dissolve various impurities in the sand. Next, the sand was washed using distilled water until the washing water had a neutral pH. The sand powder is then dried. The purified white sand was analyzed using FTIR and XRF.

3. Extraction of Silica Powder

The extraction process is carried out using the alkaline fusion method, which is to dissolve 5 g of purified sand using 60 mL of 5 M NaOH (base) and stirrer for 4 hours and heated at 80 ºC to form sodium silica (Na2SiO3) or until a gel is formed. -white gel with the following reaction equation:

SiO_2 (s) + 2 NaOH (aq) → Na_2SiO_3 (aq) + H_2O (aq)

The gel solution was then titrated using 2 M HCl (acid) until the solution had a neutral pH, with the following reaction:

Na_2SiO_3(aq) + 2HCl (aq) → SiO_2 (s) + 2 NaCl (aq) + H_2O (aq)

After that, the sample was washed with distilled water to remove NaCl and then filtered using filter paper and dried at room temperature. The dried gel is then ground to get a powder (Metungku *et al*, 2017).

The results of the white sand extraction in the form of SiO2 gel solids were tested for FTIR.

Results and Discussion

In this study, there were two samples used, namely Sara beach sand and White Sand beach sand which were then carried out based on work procedures.

1. Identify the mineral content of the sand before it is given treatment

At this initial stage, a certain amount of sand is set aside for later sorting of impurities. After that, the sand that has been sorted is then pulverized with a mortar and pestle. The fine sand was then sieved through a 40 mesh sieve.

2. Sand sample preparation

At this stage, each sample was washed with distilled water, then dried at room temperature. After drying, the sample is separated from impurities. Then the sample was mashed with a mortar and pestle, then sieved with a 40 mesh sieve to produce sand powder.

3. Identification of Mineral Content

At this stage the fine sand powder was weighed as much as 50 grams and then soaked with HCl at various concentrations, namely 2M and 3M as much as 100 mL for 14 hours. Soaking sand with HCl aims to remove impurities contained in the sand.

Fig 1: Process of soaking sand with HCl (a) soaking sand with HCl at a concentration of 2M

From the picture above, it is clear that the sand soaked in HCl with a concentration of 3M produces a solution that is more cloudy in color and there are many impurities that are lifted up. Unlike the case with 2M HCl.

Furthermore, after soaking for 14 hours, the sand was then washed with distilled water to remove the acidic nature of the sand. Washing with distilled water was stopped until the washing water had a neutral pH. After the sand washing water has a neutral pH, the sand is then filtered and dried at room temperature. The dry sand powder was then analyzed using FTIR to determine the mineral content of the purified sand. Identification of initial mineral content

FTIR Analysis

1. Sara Beach Sand HCl 2M

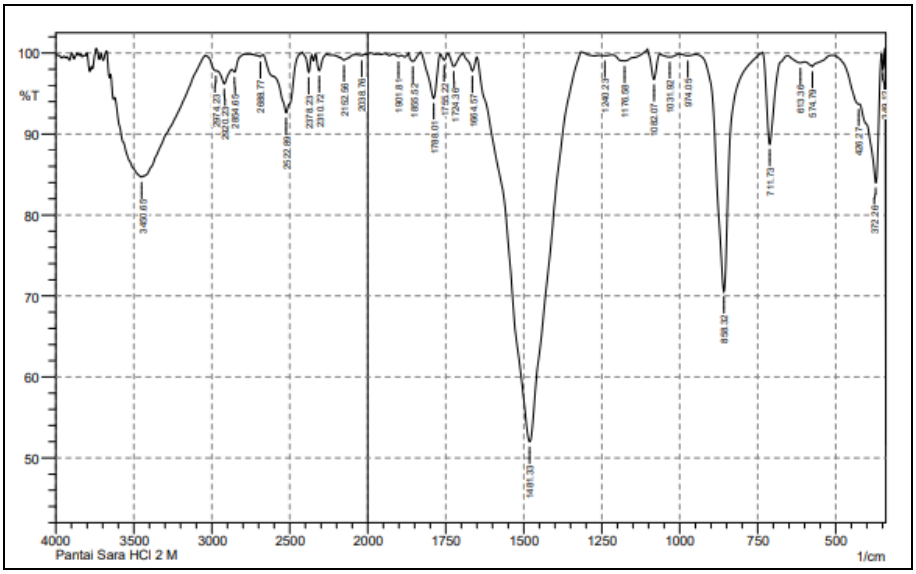

Fig 2: FTIR spectra of Sara beach sand with 2 M. HCl immersion

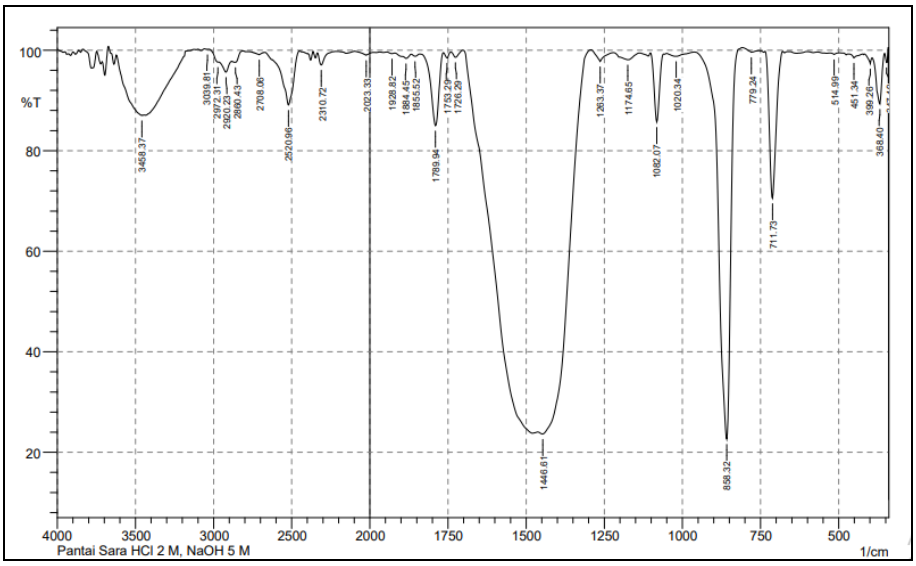

Fig 3: Spektra FTIR Sara Beach Sand HCl 2M extracted by alkaline fusion method

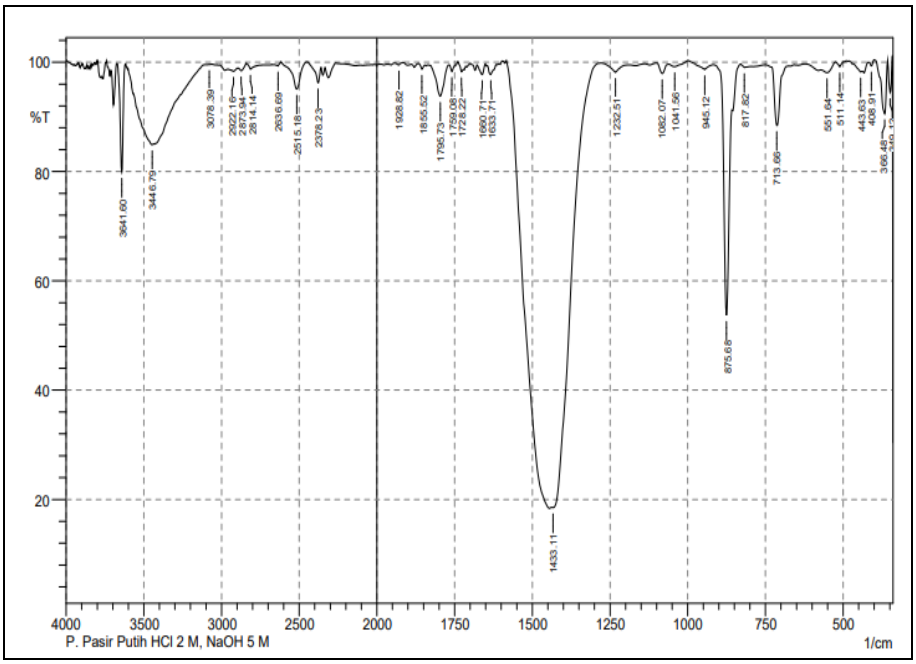

Fig 4: FTIR spectra of Sand white beach sand with 2 M. HCl immersion

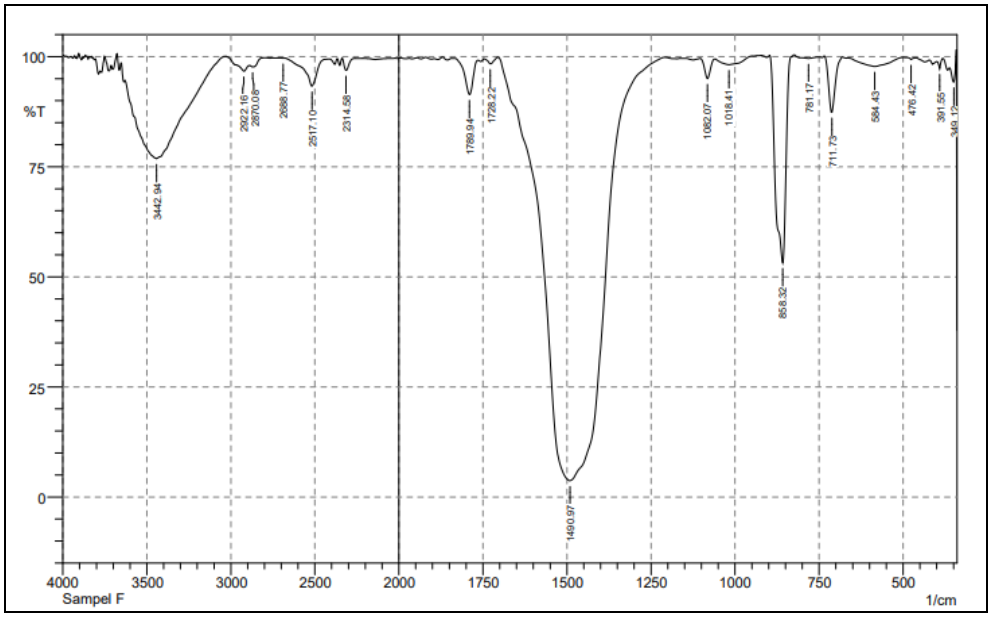

Fig 5: Spektra FTIR Sand White Beach Sand HCl 2M extracted by alkaline fusion method

FTIR analysis for the determination of the functional group of a compound in the sand. The functional groups here are focused on the silica content in the sand. Determination of the functional group of a compound can be seen at the peak that appears in a certain wavelength region based on the vibrational absorption pattern of the compound. Characterization with FTIR was carried out in the wave number range of 400-4000 cm^{-1}.

Table 1: absorption band analysis data on the FTIR. Spectrum

Frequency (cm^{-1})	Functional groups	Wave Number (cm^{-1})				Source
		Sara HCl 2M(a)	Sara HCl M (b)	Sand white HCl 2M (a)	Sand white HCl 2M (b)	
500-400	bending vibration of the siloxane group (≡Si-O-Si≡) (≡Si-O-Si≡)	426.27	497.63	470.63	428.2 466.77	Juni *et al*, 2012
680-850	Symmetric stretching vibration of Si-O in siloxane (≡Si-OH) (≡Si-O-Si≡)	711.73	711.73	711.73	711.73	
1000-900	Si-O Stretch Vibration on silanol (≡Si-OH)	974.05	977.91	-	948.98	Silverstein, 2005
1100-1000	Asymmetric Stretch Vibration of Si-O in siloxane (≡Si-O-Si≡)	1031.92 1082.07	1082.07	1082.07	1008.77 1058.92 1082.07	Adam *et al*, 2010
1800-1600	-OH bending vibration of silanol (≡Si-OH)	1664.57 1724.36 1755.22 1788.01	1633.71 1666.5 1726.29 1755.22 1789.94	1668.43 1728.22 1789.94	1728.22 1755.22 1789.94	Silverstein, 2005
2070-2090	Monohydride (H-Si-Si-H)	-	2071.55	-	-	Abuhassan, 2010
2200-2500	Si-O bending vibration of siloxane (≡Si-O-Si≡)	2310.72 2378.23	2312.65	2254.79 2353.16 2387.87	2233.57 2312.65 2384.02	Astuti dkk, 2012
3750-2800	-OH bending vibration of silanol (≡Si-OH) and H_2O	2854.65 2974.23 3415.93 3450.65	2852.72 2918.3 3439.08	2854.65 2918.3 3439.08	2854.65 2918.3 2976.16 3062.96 3437.15	Kalapathy *et al*, 2000

Silica absorption patterns that appear generally are silanol (≡Si-OH) and siloxane (≡Si-O-Si≡) groups (Trivana *et al*, 2015). Based on the data in the table above, there are several absorption bands in wave numbers that show the same results from the four samples. At a wave number of 711.73 cm-1, it shows a symmetrical stretching vibration of Si-O on siloxane (≡Si-O-Si≡) in the four samples with their respective intensities, namely, Sara beach 2M HCl 88,778, Sara beach 3M HCl 85,933, White Sand beach with 2M HCl 78,103 and White Sand beach with 3M HCl 63,757. As for the results of the bending vibration of the siloxane group (≡Si-O-Si≡) each on the Sara HCl 2M beach sand at wave number 426.27 cm-1 with an intensity of 93,705

The absorption band that appears at 974.05 cm-1 on the Sara beach 2M HCl; 977.91 cm-1 on the Sara beach with 3M HCl and 948.98 cm-1 on the White Sand beach with 3M HCl showed stretching vibrations of Si-O on silanol (≡Si-OH), but these vibrations did not appear on the White Sand beach with 2M HCl. The asymmetric stretching vibration of Si-O on siloxane (≡Si-O-Si≡) showed the same results for

the four samples, which were at the same wave number, namely 1082.07 cm-1. In addition to appearing at the same wave number, other absorption bands also appeared for wave numbers of 1031.92 cm-1 on Sara beach HCl 2M, 1008.77 and 1058.92 cm-1 on Pasir Putih beach 3M HCl.

The -OH buckling vibration of silanol (≡Si-OH) is shown by some similar results for all four samples. The absorption band at the same wave number is 1782.22 cm-1 at Pasir Putih beach HCl 2 and 3 M. In addition, at wave number 1755.22 cm-1 at Pasir Putih beach HCl 3 M and at Sara beach 2 and 3M. Absorption bands that appear at wave numbers 1789.94 cm-1 on the White Sand beach HCl 2 and 3 M and on the Sara beach sand 3M.

The absorption band that only appears in one type of sample, namely Sara 3M beach sand, shows Monohydride (H-Si-Si-H) with a wave number of 2071.55 cm-1 with an intensity of 98.756.

The Si-O bending vibration of siloxane (≡Si-O-Si≡) is indicated by the same wavenumber appearing at 2312.65 for Sara beach sand and 3M HCl and 3M HCl white sand beach sand, respectively. while the absorption band that emerged from the Sara HCl 2M beach sand sample at wave numbers 2310.72 and 2378.23 cm-1. For the 2M HCl White Sand beach sand sample, the absorption band appears at wave number 2254.79; 2353.16; and 2387.87 cm-1.

Absorption bands that appear at a wave number of 2854.65 cm-1 are found in samples of Sara HCl 2M beach sand and 3M HCl White Sand beach sand. The same absorption band also appears at a wave number of 2918.3 cm-1 which is found in samples of Sara HCl 3M beach sand, 2M HCl and 3M White Sand beach sand. Furthermore, the absorption band that appears at wave number 3439.08 cm-1 is found in samples of Sara HCl 3M beach sand and 2M HCL White Sand beach sand. The absorption band that appears at the wave number above shows the stretching vibration of –OH from silanol (≡Si-OH) and H_2O.

At this stage, the purified fine sand powder is extracted by alkaline fusion method using NaOH as solvent. The sand powder was weighed as much as 5 g and dissolved in 5M NaOH with a volume of 60 mL while stirring using a magnetic stirrer and heated at 80º for 4 hours. The dissolution forms a solution of Na2SiO3 or sodium silicate.

In the stirring process for approximately 3 hours, the solution thickens and at the end of the stirring time the sodium silicate solution becomes very thick. At this stage the reactions that occur are:

$$SiO_2\ (s) + 2NaOH\ (aq) \rightarrow Na_2SiO_3\ (aq) + H_2O\ (aq)$$

After the sodium silicate solution was formed, the solution was then titrated with 2M HCl until the pH of the solution became neutral. In addition to controlling pH by using universal indicators, the solution can also be seen where when the pH starts to become neutral, namely the formation of a precipitate or the solution seems to form two layers, and the color of the solution is slightly cloudy.

The data collected from the titration results were on the pure sand of Sara beach 2M HCl dissolved in 5M NaOH then titrated with 153.6 mL 2M HCl. While for Sara beach sand 3M HCl dissolved in 5M NaOH, the volume of HCl is 156.0 mL. For pure beach sand, 2M HCl is dissolved in 5M NaOH, then titrated with 2M HCl with a volume of 152.2 mL. On the pure sand of the White Sand beach with 3M HCl, the volume of HCl titrated is 153.2 mL.

The reactions that occur at this stage are:

$$Na2SiO3(aq) + 2HCl\ (aq) \rightarrow SiO2\ (s) + 2NaCl\ (aq) + H2O\ (aq)$$

After the pH of the solution was neutral, then the sodium silicate solution was filtered and then washed with distilled water to remove the NaCl formed during the titration attached to the silica gel. Then the gel solution formed is filtered again using filter paper.

Conclusions

In the FTIR spectrum after purification with HCl and after extraction by alkaline fusion method, the functional groups that often appear are silanol (≡Si-OH) and siloxane (≡Si-O-Si≡) groups with high intensity, which indicates the presence of SiO_2 in sample.

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