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Identification of Rock Characteristics Using XRF, XRD, and SEM Tests on Api Alam in Pamekasan

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ABSTRACT

Research has been conducted into the nature of geothermal rocks in Pamekasan. This place is located in Tlanakan Pamekasan. This study aims to determine the composition of mineral compounds in the soil so that fire can burst out from the soil and the soil does not melt. Three steps can be done with simple refinement and filtering methods, namely X-Ray Fluorescence (XRF), X-ray diffraction (XRD), and Scanning Electron Microscope (SEM). The results of XRF analysis show the element of Api Alam 1 is Ca 66.2%, Api Alam 2 is Si 37.4%, and in hot springs, is Si 39.6%. The XRD results show that mineral qualitatively in Api Alam 1 was in the form of $CaCO_3$ compound at 60.1% and SiO_2 at 39.9%, while in Api Alam 2, hot water sources were in the form of SiO_2 compound (>90%). SEM Results on Api Alam 1 show that the sample is almost homogeneous (the distribution of $CaCO_3$ and SiO_2 is uneven), the presence of slab-shaped clumps that identify the presence of varying grain size, and with high porosity, which indicates that the sample is partially amorphous in structure, in Api Alam 2. The Hot springs show irregular aggregations and poor homogeneity of the sample. The size of the three samples is ten µm or 10.000 nm, while PSA results showed an average size of Api Alam 1 was 509.7 nm, Api Alam 2 was 891.3 nm, and Hot Springs was 468.3 nm.

Introduction

Indonesia is a country that is rich in abundant natural potential, especially mining (minerals), including materials with oxidation content that have the prospect of application as nanotechnology materials, along with technological advances that indirectly accelerate the application of nanotechnology in the industrial field. This natural wealth is spread in various regions in Indonesia, so Indonesia must become one of the biggest nanomaterial suppliers in the global market [1]. Three essential things in the development of nanomaterials are making natural materials into nano size, characterizing the results of the material, and arranging nanomaterials into the final product, generating functions of new materials that never exist and are more useful for the future. The rapid development of nanotechnology is marked by the discovery of oxidizing materials characterized as intelligent materials such as ZnO, SiO_2, MgO, Al_2O_3, Fe_2O_3, Y_2O_3, CeO, Mn, and so on. The material's ability will be extraordinary if the size changes to (< 100) [2]. Many methods are used in research to obtain nano size and higher purity in this natural mineral, such as precipitation, coprecipitation, sol-gel, and others.

One of the natural materials in Indonesia is natural fire sand which is located in Tlanakan village, Pamekasan district. The local community also knows the natural fire sand as eternal fire and is used as a place for cooking and tourism. The local community doesn't know why natural fire sand could leave a fire that never is out, so it needs further research. Natural Fire Sand is located on the surface of dry land; this sand is also said to be unique because it can survive and not melt even though fire always comes out endlessly from the ground.

This sand will contain $CaCO_3$, SiO_2, ZnO, MgO, Al_2O_3, and Fe_2O_3. It is based on the color of the sand that is visible [3]. The formation of this color depends on the mixture in the sand. $CaCO_3$ sand is formed from the remains of bones and chemical precipitation processes. Calcium carbonate is the most stable crystalline phase and is widely used in the paint, paper, and magnetic industries. The color of this sand, in general, is white, yellowish-white, gray to black. SiO_2 sand is one of the mineral materials whose presence in nature is very abundant and can be used in various applications. However, the material is still mixed with other mineral elements. The function of this mineral is anti-corrosion, hydrophobic material, and so on. The color of this sand is generally white, but in nature, the color of this sand follows a mixture of other mineral elements.

In this research, as a preliminary study to find minerals in a natural fire, the aim is to identify rock properties in natural fire sands. The research team used mineral structure tests to determine which fires were not easily extinguished and which rocks did not melt, so the problem arose to determine what elements were present in the rock. The crystal structure and composition of mineral compounds in the soil can be determined through three stages, namely XRF, XRD, and SEM. The XRF test is used to determine the chemical components/composition contained in the stone. The characterization of the rock crystal structure is studied using XRD. The content and size of rock crystal grains are determined using SEM. Last, PSA is used to see the size of the stone material.

Experimental Method

Sampling was carried out on the surface of natural fires and hot springs with as many as three samples. The sampling uses random sampling. The resulting sample was heated using an oven at a temperature of 100 °C for 24 hours, then ground using a mortar until smooth. The refining results were then sieved using a 300 mesh sieve to produce nano-sized samples, the results of nano-sized samples were tested using XRF, XRD, SEM, and PSA tests.

Results and Discussions

Based on preliminary observations before testing and analysis showed that samples obtained from the three places, namely (a) Api Alam 1 are bluish-black, (b) Api Alam 2 is bluish brown, and (c) Hot Springs with samples that are still wet, so heat treatment is given at a temperature of 100 °C to eliminate the water content with a light brown color. The difference in sample color indicates a difference in mineral content in the three samples.

Analysis of sample content using X-Ray Fluorescence (XRF) and X-Ray Diffraction (XRD). XRF testing is widely used in rock or mineral analysis. An element can be determined directly by its existence without standards. The XRF results of the three grains of sand that have been tested are presented in Table 1 below.

Table 1 shows that the highest chemical element content for Api Alam 1 is Ca, followed by Fe and Si. At the same time, for Api Alam 2 and Hot Springs, the highest element content is much different where Si in the first sequence is followed by Fe and Al, with the highest peak percentage of the three sand sequentially is Ca 66.2% (Api Alam 1), Si 37.4% (Api Alam 2), Si 39.6% (Hot Springs). Fe from the XRF results in each sample becomes a supporter of the cause of the dark color in the sample. In Api Alam 1 with the highest aggregate Ca can be used as a base material for glass, ceramics, construction, and other industrial materials. $CaCO_3$ can be used as a corrosion-resistant material if used as a PANI/$CaCO_3$ composition structure in a high-pressure, high-temperature geothermal environment of around 150 °C [4]. As for Api Alam 2 and hot springs with the highest aggregate Si can be used as a base material in the manufacture of nanomaterials and as a valuable commercial product when done using the precipitation method [1]. If continued in more specific methods such as Sol-gel, emulsion techniques, Precipitation, and Coprecipitation [5]. with sufficiently high Fe content, it can also be used as a basis for making ferromagnetic materials [6].

Table 1. XRF Test Results from Api Alam 1, Api Alam 2, and Hot Springs

Element	Api Alam 1 (%)	Api Alam 2 (%)	Hot Springs (%)
Al	2.30	13.00	11.00
Si	10.10	37.40	39.60
P	0.70	0.56	0.61
K	1.20	4.94	3.57
Ca	66.20	6.29	6.53
Ti	1.10	2.10	2.12
V	0,05	0.11	0.11
Cr	0.07	0.11	0.10
Mn	0,31	0.33	0.36
Fe	11.50	32.30	33.90
Ni	0.00	0.04	0.00
Cu	0.09	0.12	0.12
Zn	0.08	0.14	0.09
Sr	1.50	0.00	0.59
Zr	0.20	0.00	0.00
Mo	1.60	0.00	0.00
In	2.40	0.00	0.00
Ba	0.20	0.00	0.00
Yb	0.24	0.00	0.00
Re	0.31	0.10	0.10
Rb	0.00	0.28	0.00
Eu	0.00	0.40	0.44
Pb	0.00	0.00	0.37

The three grains of sand were tested at the Advanced Mineral and Materials Laboratory of the State University of Malang on June 26th, 2019. The diffraction test was carried out at a long angle (10°-90°). It was intended to support the results of the three grains of sand XRF tests to find out the compatibility of the XRF and XRD results and the quantity of a sample.

Based on the results of the XRD test shows the crystallinity of the sample shown as Figure 1, in the picture, the crystal diffraction peaks are still partially amorphous as background and quite a several small peaks, so further analysis is needed to obtain maximum results, in this analysis using Match software by matching XRD results with the database contained in the Match software.

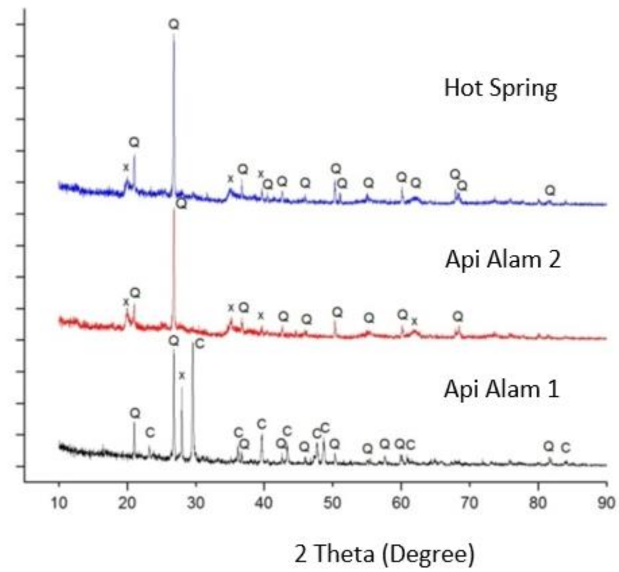
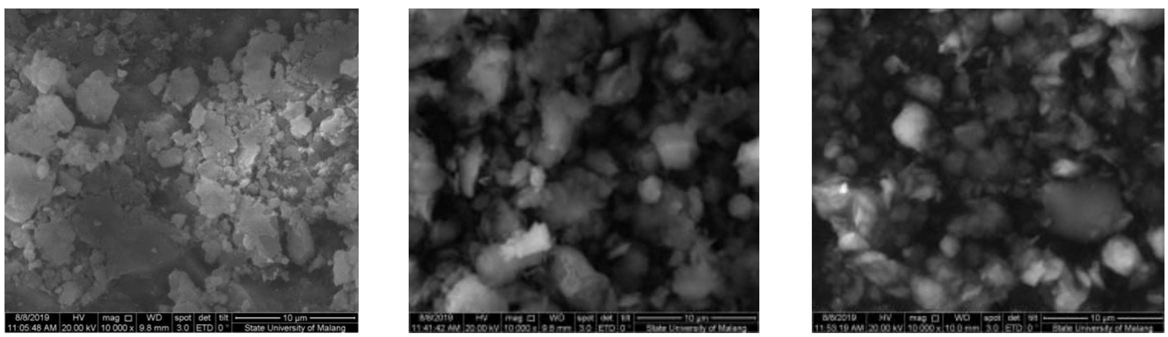


Figure 1. XRD Testing of Api Alam 1, Api Alam 2 and Hot Springs

Figure 1 shows that in the Api Alam 1 the element content is $CaCO_3$ of 60.1% and SiO_2 of 39.9%, with a peak with high intensity at theta angle 27.95 has not been identified. In Api Alam 2 the element content is Si (> 90%) with 3 small peaks that have not been identified, whereas, in Hot Springs, the element content is almost the same as Api Alam 2, namely Si (> 90%) with three small peaks that have not been identified.

Of the three samples in common, the Fe is lost at the analysis time using the Match Software. That is due to several possibilities; the first is the possibility of Fe atoms bonding with other atoms to form Pigeonite material, the second is the possibility of Fe Forming Crystals with the same structure as $CaCO_3$ and SiO_2, which both have symmetry of scalenohedral hexagonal, trigonal crystals ($3\,\bar{2}/m$), the third Fe forms an amorphous phase or low degree of crystallization such as FeO (OH) (Goetit) [7], due to the XRD results from the number of small or non-sharp peaks that identify amorphous phase formation [8]. It was also proved by the SEM results with the presence of high clumps in the form of plates and porosity [9], the fourth was quite a large number of small peaks, which made it difficult to determine the dominant peaks if manually determined diffraction peaks.

This test looks at the morphology and crystallization phase in the sample Based on Figure 2. Shows that for (a) the sample is almost homogeneous (the distribution of $CaCO_3$ and SiO_2 is uneven), it is because the synthesis process is only ordinary smoothing and sifting without heat treatment, considering the sampling site already has heat with a temperature of 167 °C and the presence of lumps that have slabs that identify varying grain size. Based on the three results, almost all sand has a high agglomeration so that the gas in the soil can come out of the soil cavity, which causes a fire. The gas is found in hot springs, which can be seen in research using geoelectricity [10] as well as with high porosity indicating a partially amorphous structural sample [11], which is a possibility the cause of fire release in the ground, as carefully by [12]. With high porosity, aggregates will accelerate the circulation of nutrients, air, and water. In (b) the sample does not show the presence of crystalline forms on silica, only visible irregular agglomerations, as well as homogeneity of the samples, which is not good. This is because the synthesis process is only ordinary sifting and sifting without heat treatment, given that the sampling site already has a temperature of 183 °C, whereas in (c) the sample shows the shape and size of agglomeration, which is the same as figure b which is where the agglomeration is irregular with porosity. Irregularly distributed unequally caused by the presence of temperature treatment only to remove the water content of a material that is a temperature of 100 °C. It indicates that some amorphous particles are still [13].



(a) (b) (c)

Figure 2. SEM Test with 10.000 x Magnification of (a) Api Alam 1, (b) Api Alam 2 and (c) Hot Springs.

Based on the three images, the sample size was 10 µm, but as a comparison, the Particle Size (PSA) was tested to determine the size of the particles. Testing 3 sand was carried out at the Physics Laboratory Material Physics Department FMIPA-ITS on August 15th, 2019. This test aims to determine particle size and particle distribution.

Table 2. PSA Test Results (d.nm)

Name	PSA Test (d.nm)
Api Alam 1	509.7
Api Alam 2	891.3
Hot Springs	468.3

This PSA test found that Api Alam 1 had a size of 509.7 nm, Api Alam 2 had a size of 891.3 nm, and Hot Springs had a size of 468.3 nm. From the results of the three different from the SEM results, all three samples have a size of 10 µm or 10,000 nm. Considering that this research still uses ordinary sifting and sifting synthesis, the possibility of natural mineral samples will be micrometer-sized. The manufacture of nanoparticles can be done physically and chemically. Physics is done by grinding in instruments such as balls mill while chemically using gas evaporation, precipitation, sol-gel, and coprecipitation methods [14]. The difference is also likely due to the influence of the ultrasonication time of sample preparation (dispersing of powder samples in water) for a long time. The length of time affects the particle size. The more ultrasonic time increases the smaller particle size and measurements using water as a medium with a dissolved sample chance that the results are read by PSA [15].

Conclusion

X-Ray Fluorescence (XRF) analysis shows that the highest elemental content for Api Alam 1 is Ca 66.2%, Api Alam 2 is Si 37.4%, and Si heat source is 39.6%. The X-Ray Diffraction (XRD) analysis shows that the mineral content is qualitatively in Api Alam 1 in the form of $CaCO_3$ compound at 60.1% and SiO2 in the amount of 39.9%, while in Api Alam 2 and hot water in the form of SiO_2 compound (> 90%) so it has the potential to be used as the basis for making nano-silica materials. Scanning Electron Microscopy (SEM) results at 10,000x magnification in Api Alam 1 show that the sample is almost homogeneous (the distribution of $CaCO_3$ and SiO_2 is uneven), in Api Alam 2 and hot springs show irregular agglomerations. The homogeneity of the sample is not good. The size of the three samples is 10 μm or 10,000 nm. Particle Size (PSA) results show that the average size of Api Alam 1 is 509.7 nm, Api Alam 2 is 891.3 nm, and Hot Springs is 468.3 nm.

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