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PREFACE

The International Mineral Processing Symposium (IMPS) series has been organized biennially since 1986, rotated among five distinguished Turkish universities: Dokuz Eylül University, Istanbul Technical University, Middle East Technical University, Hacettepe University, and Eskişehir Osmangazi University. With a history spanning more than 35 years and international recognition, the IMPS continues to serve as a platform where both domestic and international scientists and professionals in the mineral processing sector share current information, developments, and thoughts on the future of the industry. Undoubtedly, one of the main factors contributing to the symposium's longstanding success has been the support provided by both public and private sectors. We anticipate that this tradition of support will continue and grow in the same manner.

This proceeding is a compilation of 78 papers from 14 different countries presented in oral and poster sessions at the symposium. Topics included: Characterization and Mineralogy; Crushing and Grinding; Screening and Classification; Physical Separations; Flotation and Surface Chemistry; Hydrometallurgy and Bioprocessing; Concentration of Fine and Ultrafine Particles; Coal Preparation; Pelletizing, Agglomeration, and Sintering; Plant Design, Modelling, and Simulation; Industrial Minerals; Boron Minerals and Trona; Gold and Silver Processing; Rare Earth Elements (REE) and Strategic Minerals; New Processes and Technologies; Dewatering and Water Management; Recycling and Waste Processing; and Carbon Footprint Reduction.

In addition to the contributed elements of the program, we are honored to have five prominent plenary lecturers covering important topics titled:

"Optimization of Compressive Crushing"

"Understanding Classification and its Complexities in Different Applications"

"Eco-efficient Comminution Technologies"

"Enhancing Flotation Separation of Fine Particles Using Switchable Collectors"

"Anatomy of Lithium-Ion Batteries (LIBs) and their Recycling Technologies"

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M. M. Volkan BOZKURT, Halil İPEK, Kemal BİLİR & H. Serkan GÖKÇEN

Effect of sodium silicate, sodium tripolyphosphate, and sodium hexametaphosphate dispersants on particle size distribution of the halloysite, kaolinite, and quartz minerals in the dispersion process

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Abstract:

Kaolinite group clay minerals are generally formed by hydrothermal alteration of aluminosilicate minerals and have the chemical formula $Si_2Al_2O_5(OH)_4.nH_2O$. This group of ores is mainly composed of dickite, nacrite, kaolinite, halloysite, and other clay minerals, but may also contain impurities such as quartz, iron, titanium, and alunite. Halloysite is classified as harmless and a biocompatible material according to the Environmental Protection Agency (EPA 4A). However, the degree of purity determines the area where the halloysite will be used. In this respect, it is very important to determine the grade of halloysite mineral in the ore. Since halloysite mineral is chemically very similar to kaolinite mineral, it can be confused with each other in chemical and mineralogical analyses. Although the kaolinite and quartz minerals are found in the structure of halloysite ore besides halloysite mineral primarily, it is necessary to provide fine-size (-38 µm) particle liberation and to remove them from each other at these sizes to reach high purity. Within the scope of the study, the particle size differentiation of halloysite, kaolinite, and quartz suspensions was studied by dispersing in the presence of sodium silicate (SS), sodium tripolyphosphate (STPP) and sodium hexametaphosphate (SHMP) dispersants at fine particle size. In the dispersion experiments, a significant decrease in d_{90} values is observed as the dosage increases for all dispersants, while a partial decrease was observed in d_{50} and d_{10} values did not change significantly for halloysite and kaolinite minerals. In quartz mineral, the addition of dispersant did not affect the particle size importantly and d_{10}, d_{50}, and d_{90} values remained almost unchanged. Especially for halloysite and kaolinite minerals, no significant change was observed after 5 kg/ton dosage for each dispersant.

Keywords: *Halloysite, Kaolinite, Dispersion, Dispersant*

1. Introduction

Industrial raw materials are defined as rocks, minerals, or naturally occurring substances of economic value, except metallic ores, mineral fuels, and ornamental stones (Allaby, 2013). Such natural resources, which constitute an important part of daily lives, are mainly used in agriculture, ceramics, glass, refractories, metallurgy, and construction sectors; fillers, paints, fertilizers, chemicals, abrasives, gemstones, drilling muds, and electrical/electronic industries (Adekunle et al., 2015; Hamdi et al., 2018; Balde et al., 2021). In the past, pure sources found in nature could be used as they were extracted from the quarry. However, due to the depletion of pure resources, industrial raw materials are subjected to various preparation and enrichment processes nowadays (Murray, 1976; Mukherjee, 2011). In addition, with the developing analysis methods, the properties of industrial minerals can be examined in more detail and can be used in the production of new-generation nanomaterials (Zhou and Keeling, 2013; Ma et al., 2024).

Raw materials obtained from nature must be subjected to processes such as particle size reduction, classification, and mineral beneficiation to be used in related industrial fields. In this context, current

mineral processing and beneficiation methods are used in wet and dry medium. However, the selection of the mineral processing and beneficiation method to be applied is very important in order not to damage natural properties for the efficient use of such minerals in the field of nanotechnology (Takahashi, 1957; Takahashi, 1959). In addition, the processing method is of great importance in terms of ensuring particle size liberation according to the plastic and non-plastic character of the minerals. During mineral processing application, particle size reduction methods are applied in wet and dry environments in order to ensure particle size liberation. Afterward, the ore is separated into fine sizes by grinding or mixing processes depending on the plastic or non-plastic character. Mixing in aqueous media is used for the economic and technical separation of clay minerals into fine sizes. The high pulp density in this process is an important issue in terms of natural gas and water consumption in the drying stage (Wilson, 2007). The clay minerals have different effects on rheological properties during the industrial production steps (Khaydapova et al., 2015). Therefore, it is important to determine the rheological properties of clays used in wet systems. The rheological properties of clays depend on many parameters such as the solids ratio, chemical and mineralogical composition, particle shape and size distribution, dispersant type and dosage, pH, temperature, organic content, non-clay minerals, etc. (Chang et al., 1993; Maxwell and Dinger, 2008; Shoaib et al., 2022). During the dispersion of clay minerals, a small amount of inorganic or organic additives are added to the clay-water suspension to obtain a fluid mixture with high density. Such additives can be classified as deflocculants, binders, and plasticizers and are used to regulate the rheological behavior of suspensions. Sodium carbonate, sodium silicate, tetrasodium pyrophosphate, sodium tripolyphosphate, sodium polyacrylate, and sodium citrate are well-known dispersants. Binder additives are microcrystalline cellulose, ammonium alginate, and methylcellulose. Plasticizer additives are ethylene glycol, and glycerol (Tadros, 2005). Dispersion is the process by which solid colloidal particles move away from each other in the pulp such that they remain in suspension by repulsive electrostatic motion caused by the dispersant. Therefore, dispersants increase the fluidity of the suspension and reduce the apparent viscosity in aqueous suspensions (Landrou et al., 2018).

Today, understanding the fine structures of clay minerals is becoming increasingly important for the production of nanotechnological materials. Clay minerals have recently started to be used in the field of nanotechnology with various next-generation analysis methods and the unexplored properties of nano clay minerals have encouraged researchers to work on this subject. In this study, it was aimed to understand the particle size distribution of halloysite, kaolinite, and quartz suspension by dispersing in the presence of sodium silicate (SS), sodium tripolyphosphate (STPP), and sodium hexametaphosphate (SHMP).

2. Materials and methods

2.1. Materials

In this study, pure halloysite and quartz samples from Türkiye and pure kaolinite sample from Ukraine were used for dispersion experiments. The results for the mineralogical (XRD) analyses of the pure samples are shown in Fig. 1. As seen in Fig. 1a, the halloysite was composed of metahalloysite and halloysite, besides kaolinite and quartz (Fig. 1b and c) were formed as pure minerals.

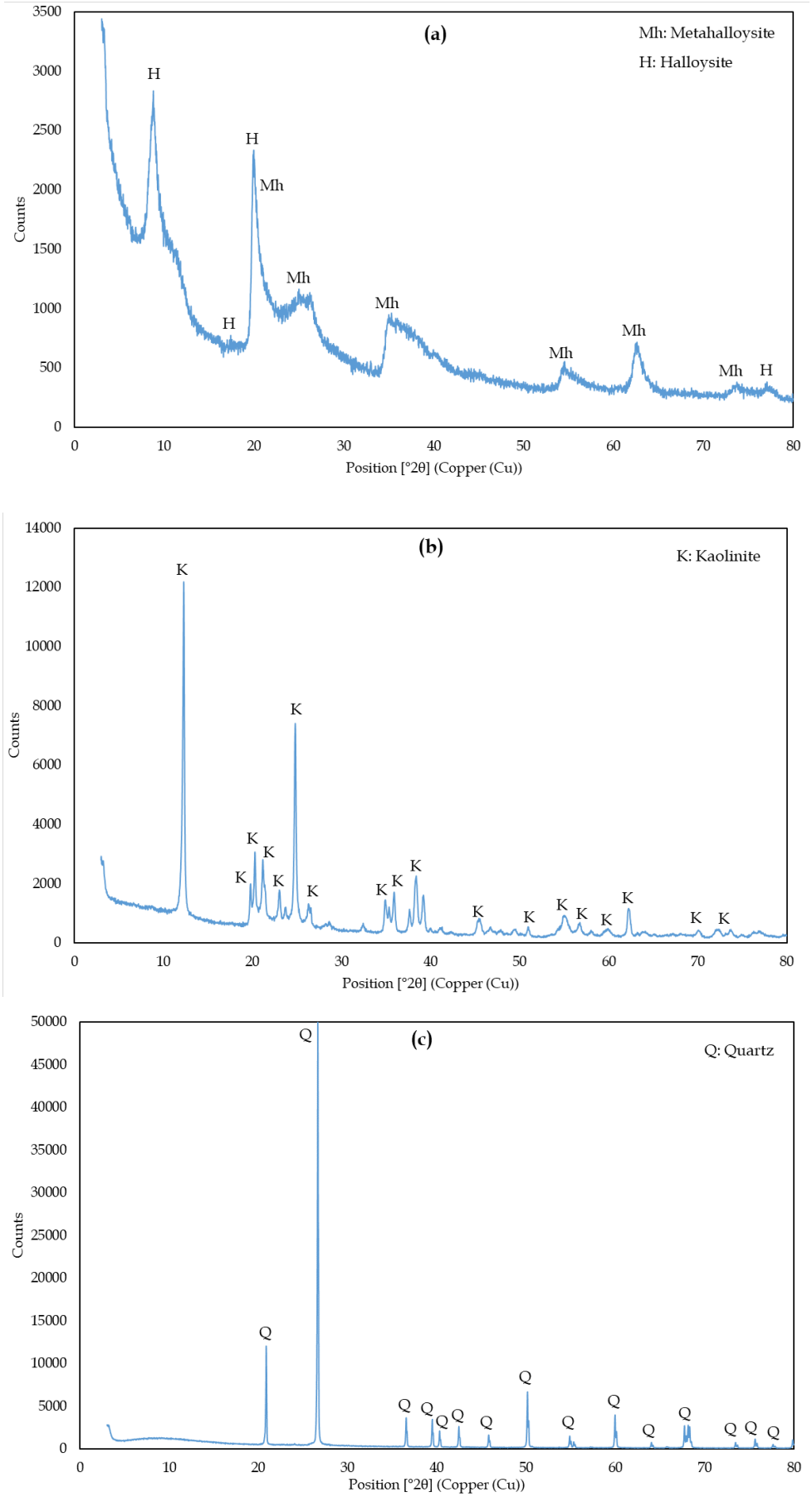


Fig. 1. The XRD patterns of (a) halloysite, (b) kaolinite, and (c) quartz samples

2.2. Dispersion experiments

Sodium silicate (SS) with a molecular weight of 184.04 g/mol and a density of 1.526 g/cm ^{3} obtained from Zag Kimya, sodium tripolyphosphate (STPP) with 94% purity obtained from TEKKİM Kimya, and extra pure sodium hexametaphosphate (SHMP) with a total phosphate content of 65-70% were used to perform the dispersing process of pure halloysite, kaolinite, and quartz samples. The experiments were carried out using dispersants at different dosages (2.5 kg/ton, 5 kg/ton, 10 kg/ton) for 30 min at 20% solids ratio in a magnetic stirrer operated at 600 rpm mixing speed. At the end of the experiment, the products were sieved with a 74 µm aperture sieve and the sieved products were analyzed in terms of particle size with a Malvern Mastersizer 2000 laser measuring device. Mastersizer measurements were performed by taking 5 drops with a pasteur pipette after the suspensions prepared at 5% solids ratio were mixed and homogenized in a magnetic stirrer for 5 min. The experimental setup for the dispersion experiment is shown in Fig. 2.

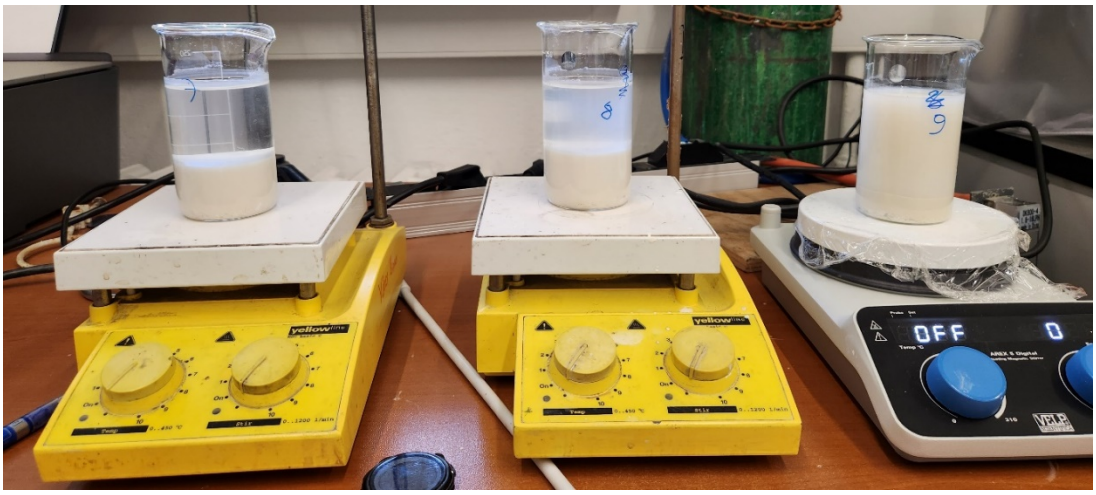


Fig. 2. Experimental set-up for dispersion experiments

3. Results and discussion

The d_{10}, d_{50}, and d_{90} values of the products of pure halloysite sample obtained from the dispersion processes at different dosages of selected dispersants (SS, STPP, SHMP) (2.5 kg/ton, 5 kg/ton, 10 kg/ton) are presented in Table 1 and shown in Fig. 3.

Table 1. d_{10}, d_{50}, d_{90} values of the products obtained by dispersing pure halloysite mineral at different dispersant dosages

	SS			**STPP**			**SHMP**		
Dosage (kg/ton)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
0.0	3.2	27.8	74.8	3.2	27.8	74.0	3.2	27.8	74.0
2.5	3.1	21.7	74.0	2.8	21.7	64.0	2.6	21.7	63.5
5.0	2.5	18.1	60.1	2.7	17.6	61.6	2.3	15.0	50.8
10.0	2.6	19.6	63.8	2.7	16.5	57.5	2.3	15.8	54.7

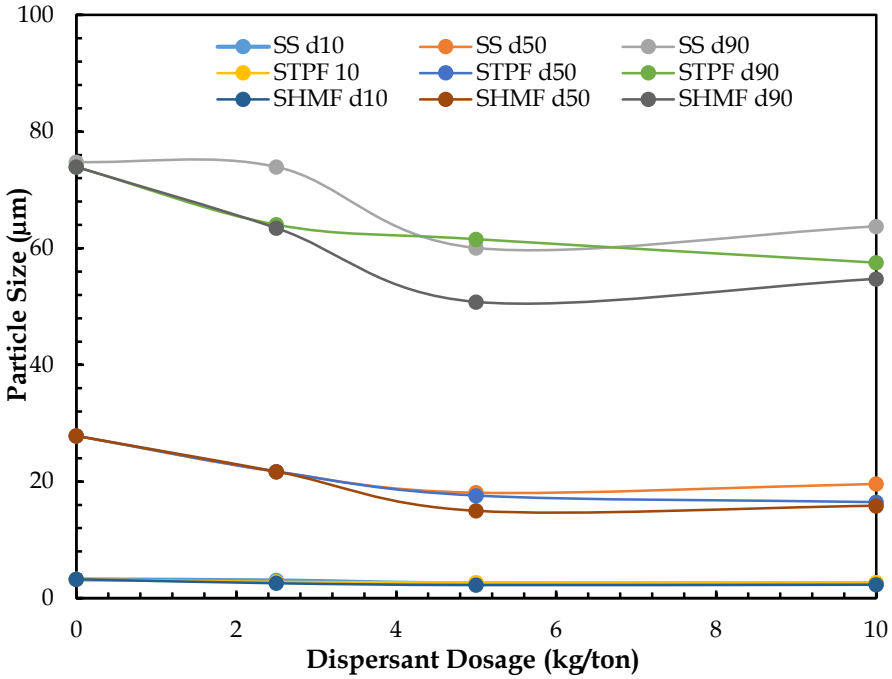


Fig. 3. d_{10}, d_{50}, d_{90} values of the products obtained by dispersing pure halloysite sample at different dispersant dosages

As d_{90} values were examined, the size value of pure halloysite sample without dispersant addition was 74.0 µm, while it decreased to 63.8 µm in the presence of 10 kg/ton SS, 57.5 µm in the presence of 10 kg/ton STPP, and 54.7 µm in the presence of 10 kg/ton SHMP. When d_{50} values were examined, while the size value of pure halloysite sample without dispersant addition was 27.8 µm, it decreased to 19.6 µm in the presence of 10 kg/ton SS, 16.5 µm in the presence of 10 kg/ton STPP and 15.8 µm in the presence of 10 kg/ton SHMP. When d_{10} values were examined, the size value of the dispersed pure halloysite sample without dispersant addition was 3.2 µm, while it decreased to 2.6 µm in the presence of 10 kg/ton SS, 2.7 µm in the presence of 10 kg/ton STPP and 2.3 µm in the presence of 10 kg/ton SHMP (Fig. 3).

The d_{10}, d_{50}, and d_{90} values of the products obtained by dispersing pure kaolinite sample at different dispersant dosages (2.5 kg/ton, 5 kg/ton, 10 kg/ton) are presented in Table 2 and shown in Fig. 4.

Table 2. d_{10}, d_{50}, d_{90} values of the products obtained by dispersing pure kaolinite sample at different dispersant dosages

	SS			**STPP**			**SHMP**		
Dosage (kg/ton)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
0.0	1.6	8.7	50.7	1.6	8.7	50.7	1.6	8.7	50.7
2.5	1.7	8.7	42.0	1.9	9.9	42.1	1.3	7.7	28.6
5.0	1.6	8.3	39.3	1.8	9.4	40.0	1.4	8.0	27.2
10.0	2.1	9.3	34.3	2.0	9.4	36.0	1.4	7.9	26.3

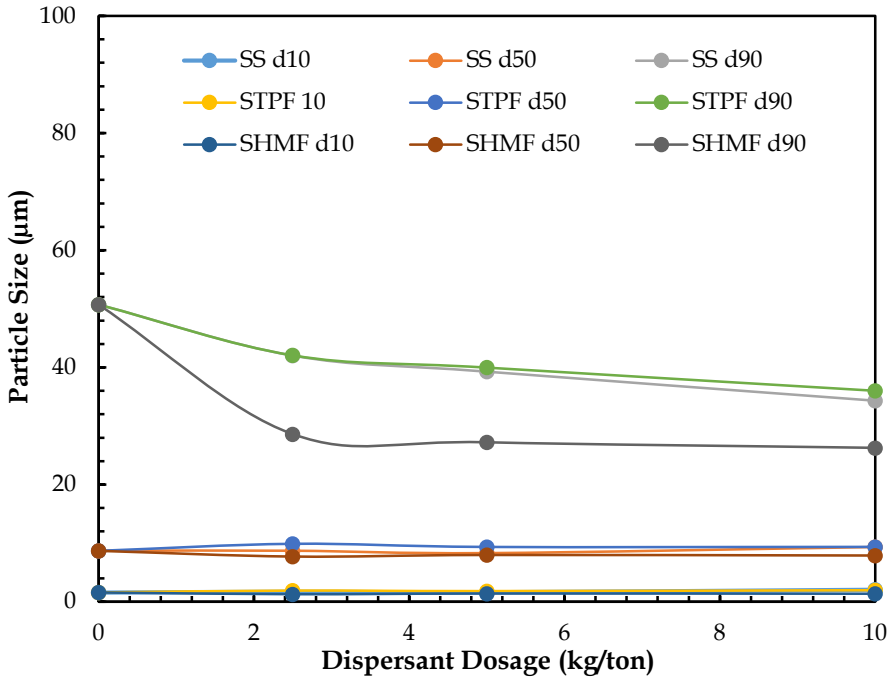


Fig. 4. d_{10}, d_{50}, d_{90} values of the products obtained by dispersing pure kaolinite sample at different dispersant dosages

As d_{90} values were examined, the size value of the dispersed pure kaolinite sample without dispersant addition decreased from 50.7 µm to 34.3 µm in the presence of 10 kg/ton SS, 36.0 µm in the presence of 10 kg/ton STPP, and 26.3 µm in the presence of 10 kg/ton SHMP. When the d_{50} values were examined, the size value of the dispersed pure kaolinite sample without dispersant addition was 8.7 µm, while it increased to 9.3 µm in the presence of 10 kg/ton SS, 9.4 µm in the presence of 10 kg/ton STPP and decreased to 7.9 µm in the presence of 10 kg/ton SHMP. When the d_{10} values were analyzed, the size value of the dispersed pure kaolinite sample without dispersant addition was 1.6 µm, while it increased to 2.1 µm in the presence of 10 kg/ton SS, 2.0 µm in the presence of 10 kg/ton STPP and decreased to 1.4 µm in the presence of 10 kg/ton SHMP (Fig. 4).

The d_{10}, d_{50}, and d_{90} values of the products obtained by dispersing pure quartz sample at different dispersant dosages (2.5 kg/ton, 5 kg/ton, 10 kg/ton) were given in Table 3 and Fig. 5.

Table 3. d_{10}, d_{50}, d_{90} values of the products obtained by dispersing pure quartz sample at different dispersant dosages

	SS			**STPP**			**SHMP**		
Dosage (kg/ton)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)	d_{10} (µm)	d_{50} (µm)	d_{90} (µm)
0.0	2.7	21.8	61.3	2.7	21.8	61.3	2.7	21.8	61.3
2.5	2.8	21.7	60.0	3.0	24.0	63.9	2.6	20.8	57.6
5.0	2.8	21.7	60.0	2.9	23.3	63.1	2.6	20.7	57.7
10.0	2.7	20.7	58.1	2.9	23.3	63.5	2.6	20.5	57.1

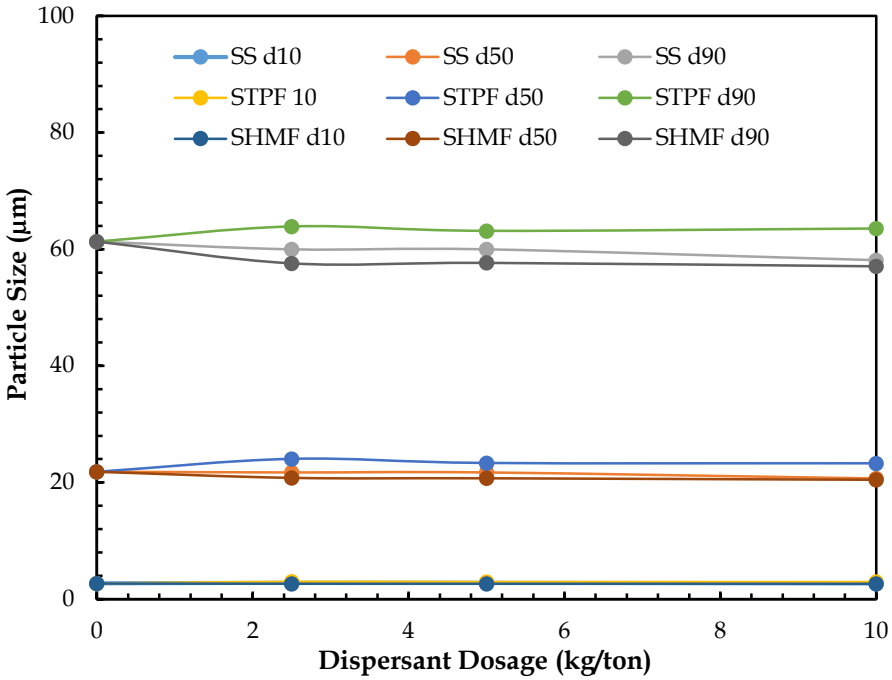


Fig. 5. d_{10}, d_{50}, d_{90} values of the products obtained by dispersing pure quartz sample at different dispersant dosages

As d_{90} values were examined, the size value of the dispersed pure quartz sample without dispersant addition changed from 61.3 µm to 58.1 µm in the presence of 10 kg/ton SS, 63.5 µm in the presence of 10 kg/ton STPP and 57.1 µm in the presence of 10 kg/ton SHMP. When the d_{50} values were examined, the size value of the dispersed pure quartz sample without dispersant addition was 21.82 µm, while it changed to 20.68 µm in the presence of 10 kg/ton SS, 23.27 µm in the presence of 10 kg/ton STPP, and 20.48 µm in the presence of 10 kg/ton SHMP. When the d_{10} values were examined, the size value of the dispersed pure quartz sample without dispersant addition was 2.7 µm, it stayed permanent in the presence of 10 kg/ton SS and changed to 2.9 µm in the presence of 10 kg/ton STPP and 2.6 µm in the presence of 10 kg/ton SHMP (Fig. 5).

4. Conclusions

In the dispersion experiments, a significant decrease in d_{90} values was observed with increasing dosage in all dispersants used for halloysite and kaolinite samples, while a partial decrease was observed in d_{50} values. d_{10} values do not change significantly. In quartz mineral, the addition of dispersant did not affect the particle size, and d_{10}, d_{50}, and d_{90} values remained almost unchanged. It was observed that there was no significant change after 5 kg/ton dosage in all dispersants used for halloysite and kaolinite minerals with dispersant addition. In this context, the optimum dispersant dosage was determined as 5 kg/ton. Besides, it was understood that SHMP produced finer-sized products compared to SS and STPP at the same dosage. In conclusion, it was understood that SHMP was a stronger dispersant than STPP and SS.

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