Journal of Geological Resource and Engineering 3 (2016) 137-141
doi:10.17265/2328-2193/2016.03.004

Development of Lead Free Crystal Glass Using Silica Sand from Gong Belibis Setiu, Terengganu

Mohamad Haniza Mahmud[1], Mohd Idham Mustaffar[1] and Abdul Hadi Abdul Rahman[2]

1. Mineral Research Centre, Department of Minerals and Geoscience, Malaysia

2. Department of Minerals and Geoscience Terengganu, Malaysia

Abstract: The silica sand deposit located at km 55 Jalan Pantai Kuala Terengganu-Kuala Besut in the District of Setiu, North Terengganu consists of loose sand of fine to coarse grain size which is developed over the raised beaches of marine origin. The underlying bedrock is believed to be either metasediment of Late Paleozoic age or intrusive granitic rocks. The average content of SiO_2 is 99.3% while for the Fe_2O_3, Al_2O_3 and TiO_2, their average content are 0.1%, 0.3% and 0.2% respectively. The screen analyses show that more than 90 percent of the silica sand is within 150 to 1,180 micron size range. The total area underlain by potential silica sand is about 1.9 million sq. metres with total measured reserve about 2.6 million tonnes. According to Malaysian Standard, MS 701:1981 and British Standard BS 2975:1988, silica sand for making crystal glass should meet the grade B specification in which the purity of silica sand is not less than 99.5% with only a small amount of iron oxide (0.015%), alumina (0.05%), chromium oxide (maximum 2 ppm) and other mineral oxides. In the production of crystal glass, a front loading furnace was used and maximum melting temperature was 1400 °C. After melting process, physical evaluation on the crystal glass products were performed pertaining to complete accomplishment of melting such as existing of bubbles inside crystal glasses. In order to ensure that crystal glass quality meets the international standard, specification for crystal glass published by British standard BS 3828:1973 was taken as a guideline. Some of the parameters are density, surface hardness and refractive index. In general, silica sand from Gong Belibis, Setiu Terengganu is suitable as a raw material for making crystal glass product.

Key words: Silica sand, attrition scrubbing, crystal glass and refractive index.

1. Introduction

The silica sand deposit located at km 55 Jalan Pantai Kuala Terengganu to Kuala Besut in the District of Setiu, North Terengganu consists of loose sand of fine to coarse grain size which is developed over the raised beaches of marine origin. The underlying bedrock is believed to be either metasediment of Late Paleozoic age or intrusive granitic rocks [1, 2]. The average content of SiO_2 is 99.3% while for the Fe_2O_3, Al_2O_3 and TiO_2, their average content are 0.1%, 0.3% and 0.2% respectively. The total area underlain by potential silica sand is about 1.9 million square metres with total measured reserve about 2.6 million tonnes. According to Malaysian Standard, MS 701:1981 [3] and British Standard BS 2975:1988 [4], silica sand for making crystal glass should meet the grade B specification in which the purity of silica sand is not less than 99.5% with only a small amount of iron oxide (0.015%), alumina (0.05%), chromium oxide (2 ppm) and titanium oxide (0.05%). Definition of crystal glass can be referred to ASTM C162-56 [5]. Crystal glass is defined as colourless, highly transparent, frequently used for art and tableware. They are heavy, glimmering and sparkling appearances as well as very exclusive and expensive. The quality of crystal glass is published by British namely as crystal glass specification, BS 3828:1973 [6].

Corresponding author: Mohamad Haniza Mahmud, M. Sc., research fields: Silica sand processing and glass ceramic.

2. Methodology

2.1 Field Investigation

Fig. 1 shows the area study of silica deposit. The area underlain by potential silica sand is about 138.5

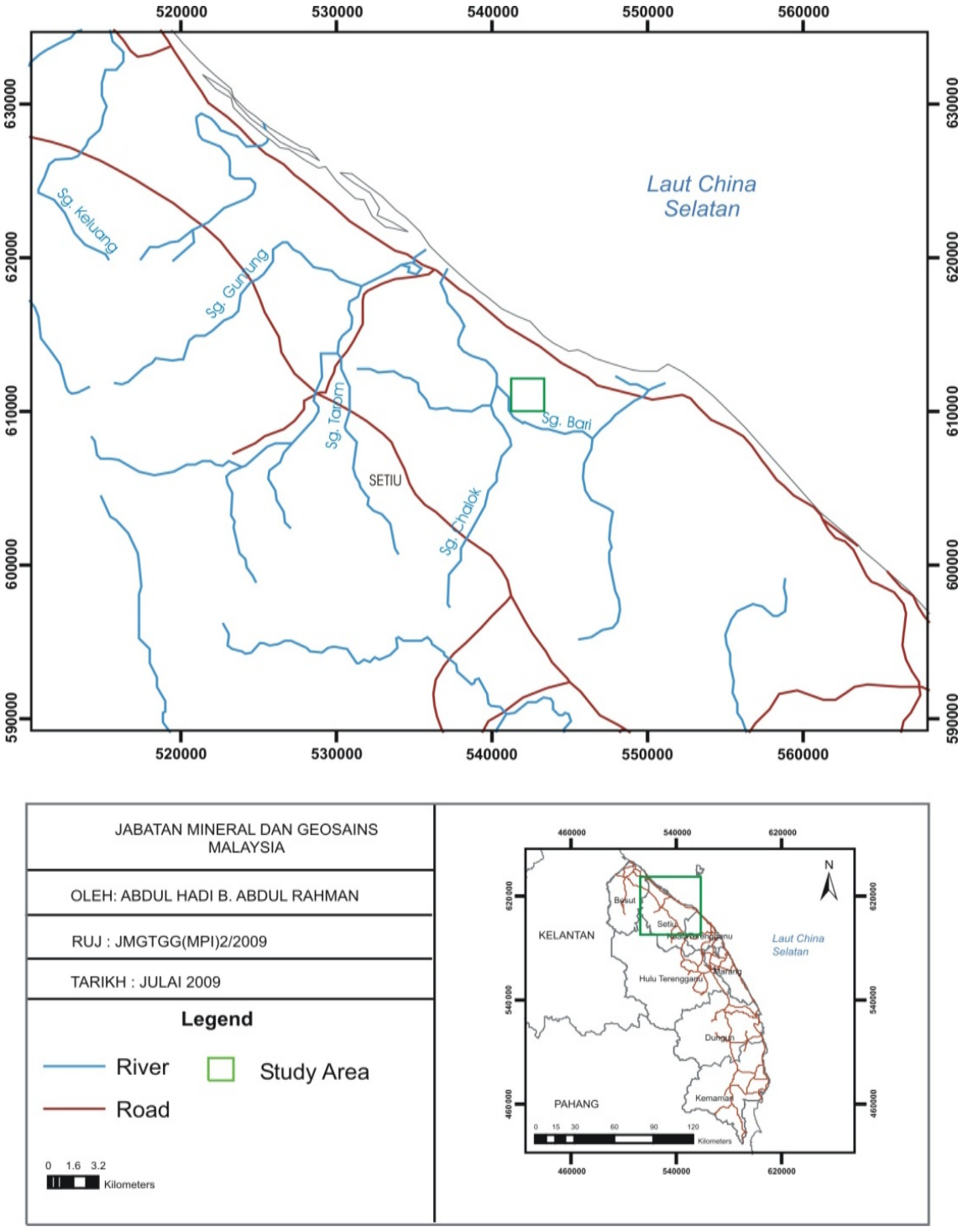

Fig. 1 Location map of the Gong Belibis deposit, Setiu, Terengganu, Malaysia.

hectares. A total of 149 auger holes were studied on a grid spacing of 100 m. Generally, three layers of sand could be classified during the augering conducted. The thickness of the overburden (upper layer), which consists of brownish grey sand, medium to coarse grain mixed with organic materials ranges from 0-0.3 m with the average thickness is 0.1 m. The total volume of overburden is 120,000 cubic metre or an equivalent of 168,000 tonnes. The thickness of the silica sand layer ranges from 0.4-3.1 m with the average of 1.7 m. The

total volume of the silica sand is 1.86 million cubic metre. Assuming the density of silica sand to be 1.4 g/cm^3, the deposit has a total measured reserve of 2.6 million tonnes. Third layer is layer of grayish brown to brown sand. The underground water table varies from 0.5-1.5 m depending on the climate.

2.2 Experimental Work

In the study as much as 50 kg of silica sand was obtained from Gong Belibis area Setiu, Terengganu. Equipment employed in upgrading the silica sand was attrition scrubber. The purpose of attrition scrubbing was generally to clean the surface of silica sand grains. It was very effective in removing ion staining [7]. After processing, the sample was analysed by X-ray fluorescence (XRF). The purpose of the sample being analysed using XRF is to identify the element composition such as SiO_2, Fe_2O_3, Cr_2O_3 and Al_2O_3. The silica product produced after upgrading process, it was ground by ball mill with agate as grinding media for approximately one hour and followed by mixing the ground silica with other chemical oxides.

In the study, crystal glass formulation is indicated in Table 1 which chemical composition involved was sodium oxide (Na_2O), calcium oxide (CaO), potassium oxide (K_2O), barium oxide (BaO) and so forth. Currently, the raw crystal glass in the market is in the form of pellet. Therefore, pelletization process needs to be carried out using pelletizer in order to get such product. Pelletizing is defined as an agglomeration process whereby an amorphous mass of finely divided particulates, such as dust, powder, is formed into a pellet, a ball or a granule in the presence of moisture added during the process [8].

Glass melting is the thermal process by which the components are completely converted into molten glass. The melting temperature and time consumed for melting depend on the type of glass to be melted [9, 10]. In this study, the time taken for melting crystal glass pellet was around ten (10) hours.

In theory, the melting process can be divided into three (3) stages namely melting and dissolving of batch materials, fining of the glass and getting it free from bubbles and seeds and lastly homogenizing the glass and getting it ready for forming. As recommended by patent No. US 6391810 B issued in 2002, the glass melting temperature is about 1,400 °C for raw material glass [11].

After melting process, casting technique was conducted to produce the crystal glass products in the desired shape and subsequently the products were stored into annealing kiln for ten to twelve hours. Annealing process occurs at around 620 °C. The aim of the annealing process was to remove internal stresses in the glasses and they would not crack or break when putting at room temperature [9, 10]. In this study, tunnel kiln was used for annealing process of crystal glass product.

In order to ensure that crystal glass quality meets the international standard, specification for crystal glass published by British standard BS 3828:1973 was taken as a guideline, Table 2 [5]. Some of the parameters are refractive index, density, and surface hardness.

3. Results and Discussion

3.1 Grain Size Distribution

Weighted amount of 20 dry selected washed samples was screened through various meshes sieve size for grain size distribution analysis. The average result of the analysis was presented in Table 3. The screen analyses show that more than 90% of sand is within 150 to 1,180 microns size range which means in the range of fine sand and very coarse sand.

Table 1 Crystal glass formulation.

Component	SiO_2	Na_2O	K_2O	BaO	B_2O_3	CaO	Sb_2O_3	Al_2O_3	Na_2SiO_3
Percentage	60-65	11-14	6-8	6-8	1-4	7-10	1-3	1-3	2-5

Table 2 Crystal glass specification, BS 3828:1973.

Category	Properties			
	% Metallic oxide	Density	Refractive index	Vickers hardness
Full lead crystal 30%	PbO $\geq$ 30%	$\geq$ 3.00	$\geq$ 1.545	-
Lead crystal 24%	PbO $\geq$ 24%	$\geq$ 2.90	$\geq$ 1.545	-
Crystal glass	ZnO BaO PbO K_2O alone or together, > 10%	$\geq$ 2.45	$\geq$ 1.520	550 $\pm$ 20
Crystal glass	BaO PbO K_2O alone or together, > 10%	$\geq$ 2.40	$\geq$ 1.520	550 $\pm$ 20

Table 3 Average grain size analysis of the Gong Belibis silica sand deposit.

Sieve (mm)	Average weight (mm)	Percentage weight (%)	Grain size category
Residue on 2.36	0.48	0.32	Gravel
Residue on 1.18	9.88	6.57	Very coarse sand
Residue on 0.6	44.38	29.53	Coarse sand
Residue on 0.3	64.95	43.21	Medium sand
Residue on 0.15	28.99	19.29	Fine sand
Residue on 0.063	1.62	1.08	Very fine sand
Passing on 0.063	-	-	Silt
Total	150.30	100.00	-

Table 4 Result of refractive index, density and microhardness.

Category	Refractive index (nD)	Density (g/cm^3)	Vickers hardness (Hv)
Sample A	1.5409	2.675	517
Sample B	1.5435	2.629	513
Sample C	1.5392	2.666	520

3.2 Physical and Mineralogical Identification

The QME (Quantitative Mineral Estimation) study shows that all of the samples consist almost entirely of quartz with trace amount of heavy minerals such as *andalusite, leucoxene, tourmaline, rutile and zircon.* The heavy minerals found in the samples analyzed were comparatively equivalent to the study by Aw [1, 2]. The quartz was mainly colorless with minor white and milky ones. The shape of the quartz grains were sub-rounded to sub-angular. Some of the quartz grains were coated partly by dark colored iron oxide [1, 2].

3.3 Characterization of Crystal Glass

3.3.1 Refractive Index

As formulated in Table 4, the refractive index of the three (3) sample crystal glass namely sample A, sample B and sample C were 1.5409, 1.5435 and 1.5392 respectively. The readings were high and this means that the quality of the crystal glass products produced from silica sand of Gong Belibis is comparable with crystal glass standard as stipulated by BS 3828:1973.

3.3.2 Density

As indicated in BS 3828:1973, shown in Table 2, there are four (4) values of density based on their percentage of metallic oxides. The full lead crystal, 30% lead oxide, where its density is greater or equal to 3.00 g/cm^3 and the lowest is 2.40 gm/cm^3 for crystal glass contains greater or equal 10% of lead oxide.

Results of density tests are summarized in Table 4. The readings of the density of crystal glass were in the range of full lead crystal and crystal glass containing less or equal 10% of BaO, PbO and K_2O alone or together.

In other word, crystal glass produced from processed silica sand is comparable with crystal glass specification, BS 3828:1973. According to Shelby [9], the difference in density value is due to many factors such as structure, bonding and chemical composition of the glass material. Lead oxide could increase of density of the glass. Other contribution factor is the cooling rate of the glass. In general, faster cooling rate yields lower density because the melt has less time to densify its structure before freezing.

3.3.3 Vickers Hardness

Results of hardness for sample A, sample B and sample C are shown in Table 4. From the results, it can be concluded that the hardness of crystal glass products produced using Gong Belibis silica sand was lower when compared with value as stipulated by British standard. The reading of crystal glass given by BS 3828:1973 is 550 $\pm$ 20 Hv. This means that crystal glass produced from such place was comparatively softer. Generally, this kind of crystal glass was easily cut and engraved.

4. Conclusion

As a conclusion, based on the experimental works, silica sand from Gong Belibis, Setiu is found to be suitable for making crystal glass. It needs simple processing to achieve B grade silica sand. Also, in this research work, it was found that the glass composition as designed by Mineral Research Centre can produce good quality crystal glass. The application of the right melting and annealing process play an important role in determining whether the crystal glass produced is better or low in quality. Characterization study showed that some properties of the crystal glass product were comparable with BS 3828:1973, specification for crystal glass.

Acknowledgement

The authors would like to thank to the Director of Mineral Research Centre, Minerals and Geoscience Department, Malaysia, Mr. Md Muzayin Alimon and the Head of Silica Based Technology Section, Mr. Mahadi Abu Hassan for their support in writing this paper. Also thanks to the supporting staff for their assistance in conducting the research work.

References

[1] Aw, P. C. 1989. *Silica Sand Deposit at Kampong Telaga Papan, Terengganu.* Geological Survey Malaysia, Report No. Industrial Mineral 2/1989.

[2] Aw, P. C. 1978. *Silica Sand Deposits at Batu Tampin and Kampung Meraga, Kemaman, Terengganu.* Geological Survey Malaysia Report.

[3] Malaysian Standard. 1981. “MS 701:1981 Silica Sand for Glass Making.”

[4] British Standards Methods. 1988. “Sampling and Analysis of Glass Making Sands, BS: 2975-1988.”

[5] American Society Testing Material. 1965. “ASTM C 162-56 Standard Definitions of Terms Relating to Glass Products.”

[6] British standard Institution. 1973. “BS 3828:1973 Crystal Glass Specification.”

[7] Will, B. A. 1992. “Mineral Processing Technology 5th Edition.” Pergamon Press, Oxford.

[8] Mollinger., P. J. 1992. “Process for Pelletization of Powder Materials and Products Therefrom.” EP 0169382A2.

[9] Shelby, J. E. 1997. *Introduction to Glass Science and Technology.* London, UK: The Royal Society of Chemistry.

[10] Varshneya, A. K. 1994. “Fundamental of Inorganic Glasses.”

[11] Armin., L. 2002. “Lead and Barium Free Crystal Glass.” US 6391810 B1.