THE UTILIZATION OF RED GYPSUM WASTE FOR GLAZES

Rose Aini Kamarudin* and Mohd. Shahir Zakaria

Department of Engineering Sciences & Mathematic, College of Engineering, Universiti Tenaga Nasional, Km 7, Jalan Kajang-Puchong, 43009 Kajang

**Corresponding author email: rose@uniten.edu.my*

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Abstract

Red gypsum (RG) is a by-product during the extraction of titanium(IV) oxide from the ilmenite ores and it causes problems such as storing and environmental pollution. In this study red gypsum, a reddish brown semi-solid mud, which is the industrial waste of a local company in Malaysia, was investigated for use in the making of glazes for the ceramic industry. The major constituents of RG are hydrated $CaSO_4$ (70 wt %), Fe_2O_3 (30 wt %) and small amount of Al_2O_3. The total accumulation of RG in Malaysia is at least 340,000 tons per year.

It was found that the addition of up to 36 % of the RG waste was possible in the production of glazes. The glazes which contain different compositions were examined for their surface hardness properties, chemical strengths in 5 % HNO_3 and 5 % NaOH solutions and crazing behaviour at 180 ^oC under 5 bars of pressure for 2 hours.

Abstrak

Gipsum merah (RG) merupakan keluaran sampingan semasa proses pengekstrakan titanium(IV) oksida daripada bijih ilmenit dan ia menimbulkan masalah penyimpanan dan pencemaran persekitaran. Dalam kajian ini, lumpur separa pepejal bewarna merah, iaitu hasil buangan industri daripada syarikat tempatan di Malaysia, dikaji kegunaannya dalam pembuatan gerlis bagi industri seramik. Kandungan utama dalam gipsum merah ialah $CaSO_4$ terhidrat (70 % berat), Fe_2O_3 (30 % berat) dan sedikit Al_2O_3. Jumlah pengumpulan gipsum merah di Malaysia dianggarkan 340,000 ton setahun.

Daripada kajian, kami dapati penambahan gipsum merah sehingga 36 % mampu menghasilkan gerlis. Gerlis yang mengandungi komposisi berbeza dikaji dari segi ciri-ciri kekerasan permukaan, ketahanan kimia dalam larutan berasingan 5 % HNO_3 dan 5 % NaOH. Sifat keretakan halus pada suhu 180 ^oC di bawah tekanan 5 bar selama 2 jam juga dikaji.

Introduction

Industrial wastes are defined as wastes caused by industrial activities such as production, manufacturing and construction. These wastes may be toxic, ignitable, corrosive or reactive in nature. If not properly managed, they cause danger to our health and the environment. Some of these industrial wastes have found end uses. However, many eventually accumulate in landfills. With greening the environment in mind and an effort to find applications for industrial wastes, a research was undertaken to find useful applications for the RG industrial waste red. Red gypsum is a waste product formed during the production of white TiO_2 pigment from the ilmenite ores [1]. The total accumulation of RG in Malaysia is at least 340,000 tons per year [2]. RG contains mainly hydrated $CaSO_4$ (70 wt %), Fe_2O_3 (30 wt %) and very small amount of Al_2O_3 [3 - 6].

Glaze in simplified term is the glassy coating on pots or tiles. It is a thin and nearly homogenous layer composed of molten silicate mixture with a lustrous or matt appearance [7]. There are many reasons for glazing a surface and amongst them are to: provide a smooth and hygienic surface, make pots impervious, enhance and decorate the surface by providing a variation in colour and texture. Most commercial glazes have a number of constituents in them with the 3 major ones being silica, flux and refractory materials. Silica creates the glassy appearance whereas flux functions to lower the high melting point of silica which otherwise would require a high temperature (1700 ^oC) to melt it. The refractory component serves to stiffen the glaze so that the glaze does not slide off the body. In pottery and glaze making, it is commonly known that potters do not use pure and refine materials because it is not practical and economical to do so. The compounds used, therefore, are those commonly found in nature and they usually provide more than one constituent. The utilization of bauxite waste in ceramic glazes has been studied before [8]. However till date, no report on using the RG waste for producing ceramic glazes has appeared in the literature.

From our earlier research, it was found that the red gypsum waste comprised hydrated calcium sulphate, iron (II) oxide and small amount of aluminium oxide which can form a glassy phase after sintering [3]. From this point of view, the objective of the present work is the use of the red gypsum waste for producing ceramic glazes.

Experimental

Formulation and Processing of the glazes

The first part of the research was to come up with glaze formulations utilizing red gypsum.

Red gypsum as glaze

Red gypsum waste received from the TiO_2 plant in Kemaman came in the form of damp, medium sized rock-like brown solid. It was first dried at 100 ºC for 24 hours before grinded using a mortar and pestle.

Sieved RG (100 g) was placed in an electric mortar & pestle; about 150 mL of water was added to it until a consistency resembling that of pancake batter was achieved. The liquid mixture was then brushed onto 1" x 1" home-made tiles. The tiles were dried overnight at room temperature and fired in an electric furnace at 1200 ^{o}C for 20 minutes with heating and cooling rates of 3.9 ^{o}C/min.

Modified zinc oxide glaze with red gypsum

For a more systematic approach to the formulation studies, the standard zinc oxide glaze was selected and modified in which the $CaCO_3$ component was replaced by RG waste [7]. It is proposed that $CaSO_4$ upon heating will formed CaO eventually and therefore served the same purpose as $CaCO_3$ in the standard ZnO glaze.

The 35 different formulations of the standard ZnO glaze are shown in Table 1. About 150 mL of water was added to 100 grams of the dry glaze mixture to form a liquid mixture with the texture resembling that of pancake batter. The heating profile is the same as above.

Testing methods for the fitting of glaze to the body

The second part of the research involved tests performed on selected glazed tiles from amongst the 35 glaze formulations above. The aim of the tests is to study the properties of the glazes. Altogether 5 tests/analyses were performed on selected glazed tiles. These 5 selected glazed tiles were selected based on their physical appearances and content of RG in the formulations.

Hardness Test (Mohs Scratch Test)

Mohs scale of mineral hardness characterizes the scratch resistance of the material in this case the glazed surface. The Mohs scale is based on ten minerals that are all readily available except the last one, diamond (Table 2). The hardness of a material is measured against the scale by finding the softest/hardest mineral that can scratch the material. On the Mohs scale, values between 1 – 5 are considered soft surface while those values above 5 up to 10 are considered hard surface.

The glazed-tile test pieces (1" x 1" x 0.4") were cleaned and dried prior to testing. The prepared test pieces were subjected to scratch using the mineral found in the Mohs Scratch Test kit. The scratch while using as minimal pressure as possible were done starting with the mineral of lowest hardness value, which is 1 on the Mohs scale and subsequently changed to the next mineral that could inflict/cause scratch to the glazed-tile surface.

Chemical Resistance Test

The glazed tiles were subjected to two different test mediums, acid and alkali. The solutions used in this test were 5 % nitric acid and 5 % sodium hydroxide solutions.

The test pieces used for this test were of 2" x 1"x 0.4" in dimension with two holes in the upper unglazed part. This facilitates the suspension of the test pieces over the solution. The test pieces were first cleaned by immersing in acetone-filled beaker that was placed in an ultrasonic bath. The test pieces were dried and weighed to record their initial weight prior to testing.

Table 1: The 35 formulations of ZnO glazes

Formulation No.	ZnO (%)	$CaCO_3$* (%)	Potash Feldspar (%)	Kaolin (%)	Silica (%)
1	8.57	22.43	30.00	40.00	0.00
2	7.68	19.20	26.88	36.25	10.00
3	6.79	16.96	23.75	32.50	20.00
4	5.89	14.73	20.62	28.75	30.00
5	5.00	12.50	17.50	25.00	40.00
6	9.52	23.81	33.33	33.33	0.00
7	8.48	21.21	29.69	30.21	10.42
8	7.44	18.60	26.04	27.08	20.83
9	6.40	16.00	22.40	23.96	31.25
10	5.36	13.39	18.75	20.83	41.67
11	10.48	26.19	36.67	26.67	0.00
12	9.29	23.21	32.50	24.17	10.83
13	8.10	20.24	28.33	21.67	21.67
14	6.90	17.26	24.17	19.17	32.50
15	5.71	14.29	20.00	16.67	43.33
16	11.43	28.57	40.00	20.00	0.00
17	10.09	25.22	35.31	18.13	11.25
18	8.75	21.88	30.62	16.25	22.50
19	7.41	18.53	25.94	14.38	33.75
20	6.07	15.18	21.25	12.50	45.00
21	12.38	30.95	43.33	13.33	0.00
22	10.89	27.23	38.12	12.08	11.67
23	9.40	23.51	32.92	10.83	23.33
24	7.92	19.79	27.71	9.58	35.00
25	6.43	16.07	22.50	8.33	46.67
26	13.33	33.33	46.67	6.67	0.00
27	11.70	29.24	40.94	6.04	12.08
28	10.06	25.15	35.21	5.42	24.17
29	8.42	21.06	29.48	4.79	36.25
30	6.79	16.96	23.75	4.17	48.33
31	14.29	35.71	50.00	0.00	0.00
32	12.50	31.25	43.75	0.00	12.50
33	10.71	26.79	37.50	0.00	25.00
34	8.93	22.32	31.25	0.00	37.50
35	7.14	17.86	25.00	0.00	50.00

*Red gypsum replaces $CaCO_3$ in RG glaze.

Table 2: Hardness of minerals based on Mohs scale

Mohs Hardness	Mineral	Absolute hardness
1	Talc ($Mg_3Si_4O_{10}(OH)_2$)	1
2	Gypsum ($CaSO_4.2H_2O$)	3
3	Calcite ($CaCO_3$)	9
4	Fluorite (CaF_2)	21
5	Apatite ($Ca_5(PO_4)_3$(OH-, Cl-, F-))	48
6	Orthodase Feldspar ($KAlSi_3O_8$)	72
7	Quartz (SiO_2)	100
8	Topaz (Al_2SiO_4(OH-, F-)$_2$)	200
9	Corundum (Al_2O_3)	400
10	Diamond (C)	1500

The test pieces were suspended in the test medium which was static with only 2 cm of the glazed-tiles dipped in it. With a static environment, it is assumed that there would be no movement in the medium which could lead to friction or abrasion on the surface of the test pieces. The container with the test pieces was then placed in a special oven already heated to 90 ºC for a period of 48 hours. The volume of the medium was maintained at the 2 cm mark at all times. After 48 hours, the test pieces were taken out, rinsed and dried prior to weighing to record the final weight of each test pieces.

Crazing Test

Crazing is a development of fine network of cracks in the finished glaze and it occurs due to the difference in thermal expansion between body/substrate and glaze under normal condition or at high temperature and/or pressure. If the expansion (upon increase in temperature) and contraction (upon returning to normal condition) of the glaze and substrate are similar, then no crazing will be observed. These cracks maybe present when the ware is first taken out of the kiln, or they may appear after a period of time. These may be plainly visible after firing or may need enhancement with ink.

Glazed-tiles of dimensions 1” x 1” x 0.4” were first cleaned with alcohol and let to dry. The tiles were first examined for any existence of cracks by applying a special ink which would show up if any cracks existed. If the test pieces passed this initial examination only then they can be subjected to the crazing test.

The test pieces were arranged in an autoclave with the temperature of the autoclave set at 180 ^{o}C and pressure of 5 Bars for 2 hours. The autoclave was allowed to cool to room temperature and the test pieces were taken out and dried. The test pieces were again subjected to the ink test to observe for any occurrence of fine network of cracks. The tests were performed in triplicate.

Results and Discussion

Visual Examination Of Glazed-Tiles

Visual examinations on the glazed tiles were made based on the following characteristics: glossiness, smoothness, color and presence of defects and the severity of the defects.

Red Gypsum as Glaze

The tile glazed with only red gypsum in the glaze formulation before firing was brown in colour. Upon firing at 1200 ^{o}C, the surface was found to be slightly shiny, orange to dark brown in color with dark black spots present.

Modified ZnO glaze incorporating RG

The appearances of the tiles glazed with the 35 different formulations containing RG are summarised in Table 3 below. The glazes formed vary from matt to gloss finish, light to dark shades of brown, with or without pinholes and crazing (fine cracks).

Tests for the fitting of glaze to the body

Altogether 35 different formulations of glazes were prepared incorporating red gypsum. Out of these, only 5 glaze formulations were selected for further tests. The selection of the 5 tiles was based on appearance and the content of the red gypsum in the formulation. It is our intention to produce glazes with high content of red gypsum in addition to acceptable aesthetic properties. The selected glaze formulations are numbers 21, 26, 31, 32 and 33. The composition of the 5 selected glazes is given in Table 4. These selected glazes have RG content between 27 – 36 %.

Hardness Test (Mohs Scratch)

Table 5 shows the results of Mohs scratch test on the 5 selected tiles. All 5 test pieces were found to be scratched by the mineral fluorite, CaF_2, which on the Mohs scale has a hardness of 4. Hence the surface hardness of the glazed-tiles is given a value of 3.5 since they were scratched by a mineral of hardness 4. In this case the RG glaze has a surface hardness of 3.5 which is considered soft surface. Values of 5 to 10 are considered normal to hard surface structures on the Mohs scale. Referring to Table 1, all the five tiles have low content of refractory material (ie kaolin and silica) which is responsible for the hardness characteristic of the glaze. In addition the composition of the glazes in these tiles have relatively high content of flux (potash feldspar) which is responsible for lowering the firing temperature and thus may cause the glaze to be softer. The $CaSO_4$ in the RG decomposes under the firing condition to CaO which will also act as flux. The soft surface structure of tiles number 21, 26, 31, 32 and 33 can be attributed to the low content of refractory material and high content of flux in the glazes.

Table 3: Physical appearances of glazed tiles containing RG

Formulation No.	Appearance of glazed tile
1	Pale-brown, matt & smooth, homogenous without any visible defect
2	Dark brown, glossy & smooth, homogenous without any visible defect
3	Dark brown, glossy & smooth, non-homogenous, some pinholes observed
4	Brown, glossy & smooth, homogenous, no visible defect
5	Light brown, matt & rough, homogenous, no visible defect
6	Pale-brown, matt & smooth, homogenous with some pinhole defect
7	Dark brown, glossy & smooth, homogenous with one pinhole defect
8	Dark brown, glossy & smooth, non-homogenous, some crazing observed
9	Light brown, glossy & smooth, homogenous, some crazing observed
10	Light brown, matt & rough, homogenous, no visible defect
11	Pale brown, matt & smooth, homogenous, no visible defect
12	Dark brown, glossy & smooth, non-homogenous, no visible defect
13	Brown, glossy & smooth, non-homogenous, some crazing observed
14	Light brown, glossy & smooth, homogenous, some pinholes observed
15	Light brown, matt & smooth, homogenous, some pinholes observed
16	Grayish brown, matt & smooth, homogenous, no visible defect
17	Reddish brown, very smooth & glossy, homogenous, no visible defect
18	Light brown, glossy & smooth, homogenous, some crazing observed
19	Light brown, glossy & smooth, homogenous, some crazing observed
20	Light brown, matt & rough, homogenous, no visible defect
21	Grayish brown, matt & smooth, homogenous, no visible defect
22	Reddish brown, very smooth & glossy, homogenous, no visible defect
23	Brown, glossy & smooth, homogenous, some crazing observed
24	Light brown, glossy & smooth, homogenous, some crazing observed
25	Light brown, matt & rough, homogenous, no visible defect
26	Brown, glossy & smooth, homogenous, no visible defect
27	Dark brown, glossy & smooth, homogenous, no visible defect
28	Brown, glossy & smooth, homogenous, no visible defect
29	Light brown, glossy & smooth, homogenous, no visible defect
30	Light brown, matt & rough, homogenous, no visible defect
31	Brown, glossy & smooth, homogenous, no visible defect
32	Brown, glossy & smooth, homogenous, no visible defect
33	Light brown, glossy & smooth, homogenous, no visible defect
34	Light brown, glossy & smooth, homogenous, no visible defect
35	Light brown, matt & rough, homogenous, no visible defect

Table 4: Composition of 5 selected tiles for physical tests

Formulation No.	Weight Percent (wt %)				
	Potash Feldspar	RG	ZnO	Kaolin	Silica
21	43.33	30.95	12.38	13.33	0.00
26	46.67	33.33	13.33	6.67	0.00
31	50.00	35.71	14.29	0.00	0.00
32	43.75	31.25	12.50	0.00	12.50
33	37.50	26.79	10.71	0.00	25.00

Table 5: Results of Mohs Hardness Test for 5 RG Glazes

Tile No.	Mohs' hardness*
21	3.5
26	3.5
31	3.5
32	3.5
33	3.5

*Mohs Hardness on scale of 1 – 10: <5 = Soft surface; $\geq$5 = Normal to hard surface

Crazing Test

The results of this test are in Table 6. Results for both before and after heat and pressure treatments are given.

Samples with visible fine cracks when given the ink treatment will be rendered failed and therefore will not be subjected to the high temperature and pressure treatments. From Table 6, it can be seen that all the test pieces passed the ink test before the autoclave treatment. This implied that under normal condition, either the process of expansion did not occur or if it did occur the difference in expansion is too small to affect the bond between the glaze and the substrate. After the autoclave treatment, only glaze number 32 did not show crazing while the other four tiles did. This implied that at 180 ^{o}C and 5 Bars of pressure, the thermal expansion between the glaze and the substrate in four out of the five tiles are dissimilar thus resulting in the crazing observed. Tile number 32 was found to be able to withstand the applied temperature and pressure in the autoclave. With no crazing observed in tile number 32, this showed that the thermal expansion between the glaze and substrate matched. The reason for this may be found in the composition of the glaze formulation itself, where the optimum use of oxides in this case ZnO is crucial in obtaining the best fit glaze-substrate thermal expansion.

Table 6 : Results of Crazing Tests on selected Red Gypsum Glazes

Glaze No.	Before autoclave test	After autoclave test	Conclusion
	Observation	Observation	
21	No crazing	Crazing observed	Glaze unstable at high temperature and pressure
26	No crazing	Crazing observed	Glaze unstable at high temperature and pressure
31	No crazing	Crazing observed	Glaze unstable at high temperature and pressure
32	No crazing	No crazing	Glaze stable at high temperature and pressure
33	No crazing	Crazing observed	Glaze unstable at high temperature and pressure

Chemical Resistance Test

Results of the acid resistance test are given in Table 7. All the test pieces showed a slight gain in weight between 0.16 – 0.46 % when they were placed in the 5 % HNO_3 solution at 90 ^{o}C. The glaze that was affected the most is glaze number 33 with 0.46 % increase in weight. Nevertheless this gain in weight is small and can be ignored. It is worth mentioning that the silica content in glaze number 33 is the highest and the RG content is the lowest amongst the 5 glazes.

Results of the base resistance test are given in Table 8. All the test pieces showed a gain in weight between 0.69 – 1.70 % when they were placed in the 5 % NaOH solution at 90 ^{o}C. The glaze that was affected the most is glaze number 21 with 1.70 % increase in weight. These test results showed that the glazes are stable in acid medium but less stable in basic medium. The weight gain in basic medium is however not very large. No explanation can be offered at this stage as to why the glazes are more stable in acid medium compared to basic medium. The overall results of the physical tests performed to ascertain fitting of the glaze with the body is summarised in Table 9.

Good bonding between glaze and the clay body was observed in all the 5 selected tiles and this is observed in the SEM image of a cross-sectional area of all the selected glazed tiles. An SEM image of a glazed tile cross-section depicting this is in Figure 1. There is no significant difference between the glaze and the body at the boundary however, the boundary is still recognizable based on the stroke of the grains where the grain of the tile body is rough compared to that of the glaze. Furthermore, no visible bubble or opening at the boundary can be seen from the SEM image thus suggesting good bonding between the glaze and the substrate.

Table 7: Results of Acid Test on RG glazes

Glaze No.		Weight (grams)			Percentage increase in weight	
		Before	After	Difference	Individual	Average
21	1	18.0972	18.1501	0.0529	0.29%	0.32 %
	2	18.2623	18.3318	0.0695	0.38%	
	3	16.9332	16.9802	0.0470	0.28%	
26	1	18.1419	18.1757	0.0338	0.19%	0.25 %
	2	17.1588	17.2041	0.0453	0.26%	
	3	18.1708	18.2232	0.0524	0.29%	
31	1	17.1235	17.1744	0.0509	0.30%	0.16 %
	2	17.5436	17.5638	0.0202	0.12%	
	3	16.9165	16.9297	0.0132	0.08%	
32	1	19.2592	19.3141	0.0549	0.29%	0.19 %
	2	17.1154	17.1378	0.0224	0.13%	
	3	18.7557	18.7870	0.0313	0.17%	
33	1	18.6166	18.6983	0.0817	0.44%	0.46 %
	2	18.4808	18.5659	0.0851	0.46%	
	3	17.8845	17.9721	0.0876	0.49%	

Table 8: Results of Alkali Test on RG glazes

Sample		Weight (grams)			Percentage increase in weight	
		Before	After	Difference	Individual	Average
21	1	18.3185	18.6026	0.2841	1.55%	1.70 %
	2	18.2470	18.6305	0.3835	2.10%	
	3	18.8057	19.0757	0.2700	1.44%	
26	1	16.3546	16.5978	0.2432	1.49%	0.69 %
	2	17.5955	17.5975	0.0020	0.01%	
	3	17.9553	18.0590	0.1037	0.58%	
31	1	16.4504	16.4540	0.0036	0.02%	1.25 %
	2	16.7448	17.0633	0.3185	1.90%	
	3	17.5633	17.8853	0.3220	1.83%	
32	1	17.7205	18.0449	0.3244	1.83%	1.55 %
	2	18.4026	18.6982	0.2956	1.61%	
	3	18.3369	18.5608	0.2239	1.22%	
33	1	18.6666	18.9896	0.3230	1.73%	1.12 %
	2	16.5786	16.5449	0.0337	-0.20%	
	3	16.6080	16.9118	0.3038	1.83%	

Table 9: Summary of physical tests results

Test/Analysis		Results
Surface Hardness (Mohs Scratch test)		Soft surface structure
Crazing Test	Normal condition	No crazing observed
	High heat & pressure	Crazing observed
Chemical resistance	Alkali	Slight staining on surface Slight weight gain
	Acid	Very little change in weight. Glaze is stable in acid medium
SEM image		Good substrate-glaze interaction

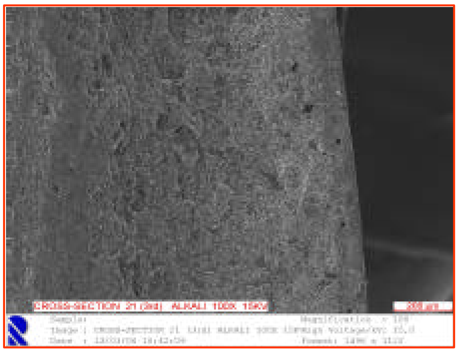

Figure 1: SEM image of glazed tile cross-section

Conclusion

Based on the above study, it can be concluded that red gypsum has potential to be used in the preparation of commercial glazes. The amount of RG utilized in the glaze formulation is up to 36 % of dry weight. The glazes produced from RG have soft surface structures (Mohs hardness of 3.5), stable under normal conditions since no crazing was observed at room temperature and pressure and stable under acidic condition but less so in alkaline condition. Hence based on these observations, the RG glazes are suitable to be used for glazing wall tiles which are not subjected to high pressure. In addition these glazes can also be used for glazing table wares.

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