

of Acid resistant materials From iron Steel making Slag

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Abstract:

This study is concerned with the feasibility of producing lining material for acid resistant applications from locally iron slag iron slag from the State Company of iron and Steel Industry at Al-Basrah is milled , sieved and tested analytically and instrumentally (i.e., X-ray diffraction) to study its composition . The examination shows that the local iron slag consists of four main compounds (i.e., Al_2O_3 , MgO , CaO , and SiO_2) besides traces of MnO , $Fe_2 O_3$, TiO_2 , $Cr_2 O_3$, and sulfur.

The experimental work is directed to treat the milled slag chemically with four steps consecutively :20% hydrochloric acid .1 % sodium hydroxide , 20% hydrochloric acid and, 1% sodium hydroxide then the remaining is treated with H_2O_2 to generate oxygen free radicals that reacts with metal sulfate to remove sulfur as H_2S . The resulting material is shaped by cold pressing under pressures ranged between 32 and 105 Mpa and three ranges of sintering temperature 950-1050 $^{\circ}C$, 1050-1150 $^{\circ}C$ and 1 150-1250 $^{\circ}C$. Acid resistivity , physical and mechanical properties of the prepared specimens are examined.

Data shows in general that at low sintering temperatures begging of liquid Phases is occurred but it doesn't give enough contact to diminish merely all the porosity. This corresponds with a decrease in bulk density , apparent porosity, compressive strength, hardness and chemical resistant whereas an increase in water absorption is noticed . In second sintering temperature rang , enough liquid phase convey all porous and consequently good impact of particles may incorporate the increase in bulk density , apparent porosity , compressive strength , hardness and chemical resistant but a decrease in water absorption is measured. Whereas ,at highest sintering temperature range , the formation of cracks

and holes in the structure is noticed because of the eruption of liquid phase that consequently decrease in bulk density, apparent porosity, compressive strength, hardness and chemical resistant, and in return, increase in water absorption is occurred.

Introduction

Many of the wastes produced today will remain in the environment for hundreds, perhaps thousands, of years. The creation of non-decaying waste materials that combined with a growing consumer population has resulted in a waste disposal crisis. One solution to this crisis lies in recycling waste into useful products. Research into new and innovative uses of waste materials is continually advancing. Many highway agencies, private organizations, and individuals have completed or are in the process of completing a wide variety of studies and research projects concerning the feasibility, environmental suitability, and performance of using recycled product in highway construction. These studies try to match society's need for safe and economic disposal of waste materials with the highway industry's need for better and more cost-effective construction materials. One of these waste materials is iron slag. (ACI, 1987).

Iron industry is one of the oldest among metal industries because it covers all fields manufacturers improve iron specification in the same time with iron slag in treatment after determination of chemical and metallurgical composition and properties. The type of slag used in many countries in the world was produced from the blast furnace the proportion of slag to iron produced by the blast furnace varies with the richness in iron of the ore and the size and efficiency of the furnace, but usually ranges from about 0.3 ton of slag per ton of iron. (Lea, 1971). The local slag that produced from the electric arc furnace is cooled slowly, but up to the present time, this slag is considered waste material. Iraqi iron industry begun from eighties and till now its by-product is considered useless in spite of huge quantities that reach 60000 ton per year. This quantity of slag throughout the plant made ahead of slag covers large area around the iron plant and consequently pollutes the environment. The slag is considered a non-metallic material which consists of SiO_2 , Al_2O_3 , MgO , CaO and some of secondary oxide such as Fe_2O_3 (Amjad, 2000).

Technological and metallurgical slag is the smelting waste in the form of dross that is separated through the smelting process, and customarily floats above the metal. In metallurgy, a suitable amount of calcium (i.e., calcium carbonate) is added to the raw material. The calcium carbonate decomposes at high degrees (900-1000 °C) to calcium

oxide and carbon dioxide. The calcium oxide, being a basic metallic oxide, combines with both silica, which is an acid oxide, and alumina, an amphoteric oxide, to produce the smelting slag in the form of calcium silicate and calcium aluminate;

The chemical composition of slag varies greatly due to the variety of metallurgy's, and to the variety of raw materials used in each metallurgy. In iron industry, raw material may take the form of ferrous oxide, ferrous sulfide, or old ferrous (scrap iron). Due to the high temperature of slag iron furnace and the availability of raw materials, mixed mineral oxides are formed in certain crystalline composition that can impart various characteristics after certain treatment.

In iron metallurgy, the iron used contains a high proportion of mud flaws that must be removed since they debase the quality of produced iron. For this reason the calcium and dolomite are used as a catalyst for smelting with coke coal to produce the required heat for smelting. In addition to carbon dioxide, that produced from the decomposition of calcium carbonate, it reacts with flaws (silica and alumina) in the raw iron to form compounds in the shape of a layer of smelting liquid floating on the surface of the smelted iron due to its low density where slag is formed. As for this type of produced iron, it is called steel iron as it contains a proportion of flaws that debase the quality of the iron (Lea, 1970; Jamel and Adjani, 1980).

The proportion of slag to steel iron varies according to the increase or decrease of the iron proportion in the raw iron, in addition to the capacity and size of the smelting raw iron furnace. This proportion ranges between 0.3 ton of slag for each 1 ton of the steel iron that produced. In 1974 world production of steel iron was about 513 million ton per year, and this in turn gave more than 250 million ton of slag (Smolczyk, 1980) in this exploitation, locally available slag from Iraqi Steel and Iron Industry of Um-Qasr is chemically treated to characterize the requirement of chemical resistant material.

Experimental Procedure :

Scheme(1) illustrates the experimental sequence that followed through this investigation. Slag from State Company of Steel and Iron Industry at Um-Qasr was used as a raw material.

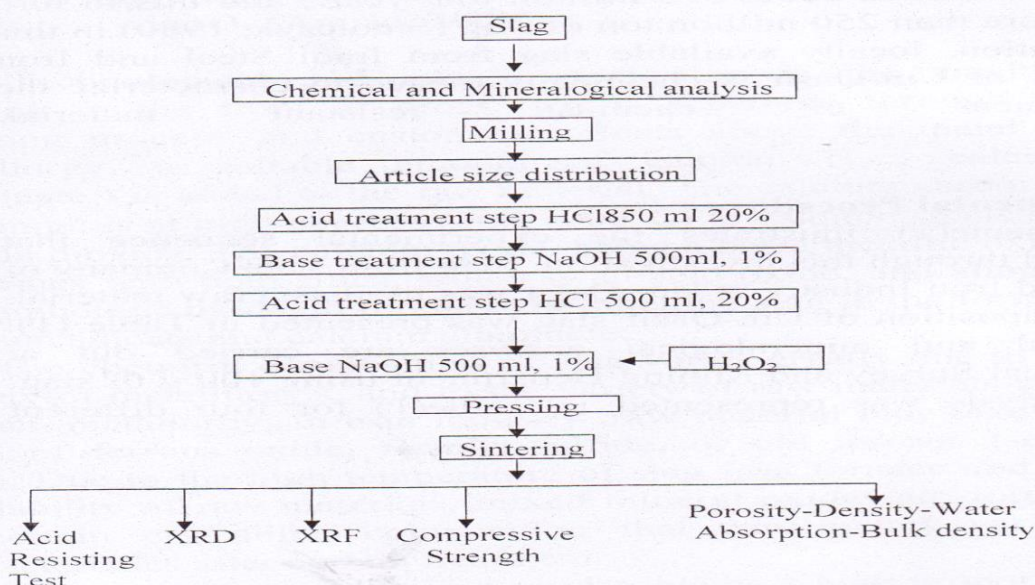
The composition of Um-Qasr slag was presented in Table (1). Chemical and mineralogical analyses are carried out at Geological Survey and Mining Department using 100 g Of slag.

The analysis was represented in Table(1) for four different samples

Table(1) Chemical Analysis Result of Um-Qasir Slag

Sample No.	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO
1	15.62	18.6	28.18	27.99	8.47
2	25.92	10.96	17.3	26.67	17.85
3	28.39	24.52	8.38	16.88	20.78
4	22.16	6.22	32.7	21.67	11

The residual components of slag consists of 2% Fe₂O₃, 4.13% MnO, 1.2% Cr₂O₃, 2% S and 2% alkali



Scheme (1) Technological routine for this investigation

Apparatus and Instrument :

The following apparatus and instruments were used in this investigation :

- 1 . Hand hammer.
2. Vibratory ball mill, type (apex 8000 mill) with carbon steel ball each of 8 mm diameter.
3. Laboratory test sieve of 125 micron Aperture.
4. Electronic balance of z|-digit accuracy Type Shimadzu libror AEU-210 capacity 200,000g made in Germany.
5. Electric mixer type Heidolph RZR 2050 electronic.
6. Electric toeplate type Stuart Scientific made in UK.
7. pH meter Type metro HM, 686TIT- processor, swiss made.
8. Furnace of max. temperature 200 C Type bicasa voutomtic Italy made.
9. Hydraulic press of 30 ton capacity Type' RIIC research and Industrial Instruments Company London-England.

10. Programmable Electric furnace Type Nabet-therln Max. Temperature (1400 °C) Model L08/14, made in Germany.
- 11 . X-ray diffraction Type (Philips) PW1840, CU Target NiFilter T=1.54,
12. X-ray fluorescent PW 1450/10 Cambridge Stereo Scan Type 240 Mix 10,000 x made in England.
13. Stereo Pycnometer, Type QUANTA-CHROME made in Germany.
14. Rockwell Hardness tester, Type Rockwell N15, Wilson- Rockwell-mod B554-T-U.S.A.
15. Compressive strength test user Type Schenck Treble compressive strength tester, RM100 Max. 100 k N , made in Germany.

Experimental Procedure:

Milling Step:

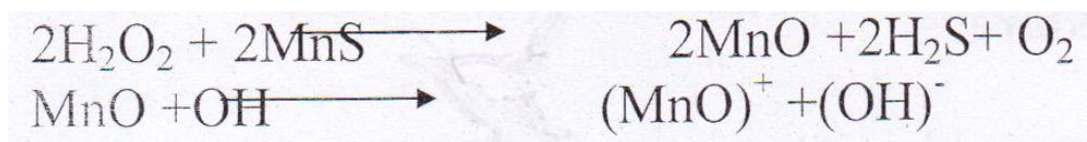
Figure 1 shows a block-diagram of the experimental steps that outlined the whole experimental program. Preliminary preparation of the slag is commenced by hammering the samples using hand hammer. After wards, the sample is endures milling using a vibratory ball mill of 62.8cm³ capacity. Milling of the sample was lasted for 3 hrs. Then, sieving by hand using (eve of 125 micron aperture finalized the preparation of the sample

Chemical Treatment Step :

The chemical step was commenced by following :

- 1 . 1 00g of prepared slag was transferred into a 1 liter flask and 850 ml of 20% concentration of Hydrochloric acid was added over the content.
2. Heating at 100 °C with vigorous mixing to guaranty good homogeneity and to get rid of any un-dissolved material.
3. Thoroughly washing using hot water into another flask of 1 liter capacity where 500 ml. of 1% of sodium Hydroxide was added afterwards to maintain basic media of the resulting solution to convert all undissolved salts to Hydroxides for subsequent stages by generating water dissolvable salts. This step was done with gentle mixing at temperature of 100 °C.
4. Afterwards the material was washed thoroughly with hot water where 500 ml of 20 % concentration of hydrochloric acid was added with gentle mixing at 100 °C where all the hydroxides reacts forming dissolved chlorides, which can be removed from the material by washing with hot water.

5. Finally 500 ml of 1 % sodium hydroxide was added till 9 pH has attained followed by addition of 10 ml of H₂O₂ to allow the resulting oxygen radical to react with the metal-sulfide (i.e. MnS) for removing the sulfur as HaS. This step was lasted for 2 hours at 80 °C with gentle mixing to allow the oxide to react solely the hydroxide ion to form metal oxyhydroxide according to following reactions:



Weight Loss Determination :

To characterize previous stages, the feasibility of chemical treatment stages were customized to meet best time of treatment in each step that correspond to maximum weight loss. In these experiments, best times were matched to 135 min for hydrochloric acid step, 45min for sodium. hydrochloric step ,90 min for second hydrochloric acid and 45 min for peroxide step that correspond to maximum weight loss of 36g, 9.2g, 15g and 5g respectively. This means, that 35g of final material was remained from 100g of the starting slag. The resulting material was filtered and dried before XRD analysis was performed.

Seemingly, four main types of mixed metal oxides were monitored. These are MnAlFeO₄, FeAl₂O₄, MgAl₂O₄ and CaAl₂Si₂O which were reasonably incorporate the acid- resistivity of the resulting material.

Specimens Pressing Step :

Herein, the resultant was shaped using dry and cold press.

The specimen was shaped into disks with a diameter of 20 mm and thickness of 5 mm.-This was performed using steel mould and hydraulic press with capacity of 30 ton. Different compaction pressures of 32, 55, 87.5, and 105 Mpa were initially experienced to determine optimum conditions. This was attained by visual inspection and later by using an optical microscope to examine the existence for any minute cracks that may occur in the prepared specimen. This compaction pressure was corresponded to 87.5 Mpa .

Sintering Step :

The sintering of the prepared specimens was carried out in a programmable electric furnace, after attaining the required maximum

temperature, the specimens was kept for 2 hours then, cooling of the specimens was commenced at a rate 3⁰C/min.

Physical and Mechanical Properties Determination :

Bulk Density and Porosity :

In the ceramic body, the density is the relation between the solid part to the gas part that considered as the most important physical property, which determined the features and types of the ceramic products. The real density was measured by Stereo Pycnometer. Measurements were performed by weighing the specimen using a sensitive electronic balance with accuracy of 0.0001 g. Density of specimens was calculated by measuring their volumes. Water absorption, size of the opened, closed porosity, apparent porosity and bulk porosity was determined after during the specimen in a furnace for one day and measuring its weigh (D). Then, specimen was immersed in boiling water for 5 hours and left in water for further 24 hour.

Reweighing as it was inside a hanged basked (S). After that the specimen was taken outside and rub with a piece of fabric, then weigh (M) was measured.

Bulk density and the apparent porosity are calculated using the following relationship (Ceram, 1989).

$$\text{Bulk Density} = \frac{D}{M-S} \quad \dots(1)$$

$$\text{Apparent Porosity}(\%) = \frac{M-D}{M-S} * 100 \quad \dots(2)$$

Accordingly, the opened and closed porosity and water absorption can also be calculated by following equations:

$$\text{Water Absorption}(\%) = (M-D) * 100 \quad \dots(3)$$

$$\text{Volume of open porosity} = (M-D)/p \quad \dots(4)$$

$$\text{Volume of close porosity} = (D-S)/p \quad \dots(5)$$

Hardness Test :

Hardness was determined as the resistance of the material surface for abrasion or scratch. Hardness was measured using Rockwell device type (Wilson-Rockwell-Mod B 554-T-U.S.A.).

Compressive Strength Test:

Compressive strength was determined as cementing value of the ceramic body and its ability to keep this cementing with the change of the uses condition. The cylinder shaped specimens were prepared with diameter of 30 mm and height of 60 mm .After sintering process, the test was made by a testing machine at pleading average of 0.5 min/min, the compressive strength is calculated by using the following relation according to the specific ASTM C773 :

$$\sigma_c = \frac{P}{A}$$

Where:
 σ_c = compressive strength (Mpa)
 P = applied pressure (N).
 A = Area loading (mm²).

Acid Resistivity Test :

This test was performed to characterize acid resistivity of the resulting specimen at various conditions. This test was commenced according to Russian specification (GOST 900-41). After drying for 24 hours in an electric dryer at 110 °C, the specimens were weighted and initial weight was recorded as W_1 .After that, the specimens were placed in a thermal glass container and immersed in hydrochloric acid solution of 10% concentration. The container was kept at 100 °C for three hours inside a water bath. After washing and drying the specimens, reweighing was recorded as W_2 This test was repeatedly performed by immersing two similar sets of specimens in both 10% HNO₃ and 10% H₂SO₄ in order to explore the chemical resistivity using different acidic solutions. The chemical resistivity was examined. This test was again performed at higher temperature 200 °C. The chemical resistivity was calculated using the following relation.

$$\text{Weight Loss(\%)} = \frac{W_1 - W_2}{W_1} * 100$$

Results and Discussion

Chemical Treatment Step :

Chemical treatment of the slag served to separate phases of less acidic resistivity chemical structures; these phases formed basically as a result of the electrical arc furnace. Data extracted from XRD analysis of final product just after chemical treatment step are summarized in Table (2). These phases are as follows:

A. single phases contains (SiO_2) B. mixed phases contains (MgAl_2O_4 , FeAl_2O_4 , MnAlFeO_4 , $\text{MnO}_3\text{Mn}_2\text{O}_3\text{SiO}_3$, $\text{Ca}_3\text{Mg}(\text{SiO}_4)_2$, $\text{CaAl}_2\text{SiO}_8$ and MnAlFeO_4).

Practically all the single and mixed phases show chemical resistance toward acid attack. These phases play a basic role in glass resistivity from both chemical and mechanical effects especially after sintering. Especially the mixed metal oxide phase of MnAlFeO_4 , since its cationic charge imparts an electron shell against acid attack.

The Influence of Sintering Step :

Sintering at different temperature ranges is commenced to investigate the proper range of temperature that impedes practical importance for the resulting material according to the studied physical and mechanical properties. Besides, to its importance in inducing new phases that ensue much of its acid resistivity against acids attack. Table (3) lists the phases that formed after sintering. These phases are classified into two types.

Stable phases: : Such as MgAl_2O_4 , FeAl_2O_4 and MnAlFeO_4 that improves the mechanical and chemical properties of the resulting material.

New phases :

Such as $\text{Mg}_2\text{Al}_4\text{Si}_5\text{O}_{18}$, $\text{CaAl}_2\text{SiO}_6$, Al_2SiO_5 and $\text{Al}_6\text{Si}_2\text{O}_{13}$. These phases generated by the action of heat sintering and because of low melting temperatures of its constituents such as Ca, Al, Mg, and Fe that result to higher viscosity of the solid solution which in return diffuses among the particles and improves both mechanical and chemical resistance of the resulting material.

Table (4) shows the composition of the sintered specimen. The test was done by laboratories of Science College of University of Mousal. Scanning electron microscopy shows the following for the three ranges of temperature, at sintering temperatures of 950- 1050 °C beginning of liquid phases enhance low imbedding between particles since not enough liquid to close all the pores.

At sintering temperature 1050-1150 °C where complete soaking of the granules by the liquid phases is occur (i.e., mix metal oxides) that result in closing all pores, and hence good impact is endured whereas, at sintering temperature 1150-1250 °C the formation of cracks and holes in the structure is occurred due to liquid phase eruption.

Acid resistivity of the sintered material was tested in the three different acids; hydrochloric, nitric and sulfuric acid each of 10% concentration, Tables (5),(6), (7), (8), (9) and (10) list the percent of weight loss or each sample. Data shows that the loss in weight was greater at temperature of sintering between (950- 1050 °C). While, at temperature range between (1050- 1150 °C), lower values were noticed. The losses become higher when temperature reaches 1250 °C because of formation of crack and holes. Herein, the increase in chemical resistivity against acid is attributed to the following reasons:

- 1 .Removing all free impurities from the slag that aggressively attacked by the acid in the course of chemical treatment stage.
2. formation of new phases not just through the course of chemical treatment but also by the action of temperature of sintering that imparts the cubic crystal structure coordinates, with the distribution of the silicon dioxide in such coordinate that enhances the formation of ionic shelled and increases the chemical stability against acids.
3. In addition to the influence of sintering temperature on forming a liquid phase that penetrates among granules and closing the pores of the resulting materials.

Effect of Sintering Temperature on Bulk Density :

Figure (1) illustrate the relation between the bulk density of specimens and sintering temperature. It appears that the bulk density values shows lower values when samples were sintered at temperature (950-1050 °C) than theoretical value as calculated. The reason has been previously around (2.3-2.5 g/cm³). reasoned as no enough liquid for pores closure, (i.e., porosity becomes higher).Hence, lower values of density were noticed. While at higher sintering temperature between (1050-1150 °C), the increases values were noticed. Theoretical values of density were noted at sintering temperature 1150 °C where best properties were notice which migt also be attributed to the existence of the spinal phase such as (MgAl₂O₄) and ceramic bonds. Wheras, at sintering temperature between (1150-1250 °C), the decrease in density values were noticed and attributed to bubbles formation of liquid phase due to excess heat that

force these bubbles to migrate and causing inconsistent in solid phases.

Apparent Porosity, Temperature and Water

Absorption :

Figures (2) and (3) examine the effect of sintering temperature on apparent porosity and water absorption respectively. Herein, the influence of temperature had matched its influence on bulk density but in reverse action. Simply, the decrease in porosity and water absorption was corresponded to an increase in bulk density and vice versa. Same reason was accommodated with the previously mentioned ones for the increase and decrease in bulk density at different sintering temperatures .

Effect of Sintering on the Compressive Strength :

Figure (4) shows the relation between the compressive strength and sintering temperature. It seems that higher compressive strength values was occurred at sintering between (950- 1150 °C), which might attributed to the existence and formation of spinal phase, which contains MgAl_2O_3 , and FeAl_2O_4 . Tese two phases are simply entered among the granules and closes all the pores and consequently increase the compressive strength. At sintering temperature between (1150- 1250 °C), lower compressive strength values were noticed because of cracks and large hole existence as a result of liquid phase evolution.

Effect of Sintering on the Hardness :

Once more, Fig (5) shows higher hardness values at sintering between (950- 1150 °C), the reason was previously related to the formation of spinal phase of MgAl_2O_3 , and FeAl_2O_4 that imparts improvement on the mechanical characteristics of the sintered specimen. Once again, the decrease between (1150-1250 °C) was resulted due to formation of cracks and large holes as a result of evolution of the liquid phase.

Table(2) XRD Data after chemical treatment

No.	Phase	d-Standard	d-Measure	I
1	NaAlSi ₃ O ₈	3.19 3.81 1.67	3.19 3.77 1.67	100 70 50
2	SiO ₂	3.58 6.00 3.87	3.55 6.00 3.84	100 80 70
3	MgAl ₂ O ₄	2.44 2.02 1.43	2.45 2.04 1.47	100 70 60
4	FeAl ₂ O ₄	2.45 2.02 1.43	2.45 2.04 1.47	100 80 80
5	MnAlFeO ₄	2.47 2.90 1.58	2.45 2.91 1.58	100 60 60
6	Ca ₃ Mg(SiO ₄) ₂	2.67 2.45 4.70	2.69 2.45 4.73	100 80 50
7	MnO ₃ Mn ₂ O ₃ SiO ₃	2.72 1.66 2.14	2.69 1.67 2.16	100 70 50
8	CaAl ₂ Si ₂ O ₈	3.19 3.18 3.21	3.19 3.18 3.21	100 90 60

XRD

Table (3)

Data for after sintering

No.	Phase	d-Standard	d-Measure	I
1	MgAl ₂ O ₄	2.44 2.03 1.55	2.44 2.02 1.55	100 70 60
2	FeAl ₂ O ₄	2.44 2.03 1.55	2.45 2.02 1.55	100 80 80
3	Mg ₂ Al ₄ Si ₅ O ₁₈	2.44 2.03 2.98	2.45 2.02 2.98	100 90 90
4	MnAlFeO ₄	2.47 2.90 1.58	2.47 2.9 1.58	100 60 60
5	CaAl ₂ SiO ₆	2.94 2.48 2.86	2.98 2.44 2.85	100 50 50
6	Al ₂ SiO ₅	3.36 2.20 3.41	3.35 2.22 3.35	100 100 90
7	(Ca, Mn)(Mg,Fe, Mn)Si ₂ O ₆	2.98 2.91 2.52	2.98 2.90 2.51	100 80 70
8	NaAlSi ₃ O ₈	3.19 3.69 1.67	3.23 3.64 1.64	100 80 60

Table (4) Composition of specimen after sintering by XRF (PW1450/10)(University of Mousal, 2003)

<u>Oxides</u>	<u>Analysis(Wt.%)</u>
SiO ₂	69.85
Al ₂ O ₃	11.56
Fe ₂ O ₃	2.73
CaO	5.09
MgO	3.68
MnO	4.13
NaO	1.57
TiO ₂	1.39

Chemical structure on oxygen atomic base

<u>Element</u>	<u>Concentration(Wt.%)</u>
Si	32.59
Al	6.10
Fe	1.90
Ca	3.64
Mg	2.22
Mn	3.19
Na	1.17
Ti	0.83
O	48.36

Table (5) Weight loss in 10% HCl at 100 °C

NO.	Sintering Temp. (°C)	Time (min)	Weight Loss (%)
1	950	180	0.9161
2	1150	180	0.4285
3	1250	180	0.6851

Table (6) Weight loss in 10% HNO₃ at 100 °C

NO.	Sintering Temp. (°C)	Time (min)	Weight Loss (%)
1	950	180	0.5730
2	1150	180	0
3	1250	180	0.2240

Table (7) Weight loss in 10% H₂SO₄ at 100 °C

NO.	Sintering Temp. (°C)	Time (min)	Weight Loss (%)
1	950	180	0.5437
2	1150	180	0
3	1250	180	0.2522

Table (8) Weight loss in 10% HCl at 200 °C

NO.	Sintering Temp. (°C)	Time (min)	Weight Loss (%)
1	950	180	1.812
2	1000	180	1.4382
3	1050	180	1.2987
4	1100	180	1.0261
5	1150	180	0.7027
6	1200	180	1.22
7	1250	180	1.604

Table (9) Weight loss in 10% HNO₃ at 200 °C

NO.	Sintering Temp. (°C)	Time (min)	Weight Loss (%)
1	950	180	0.7391
2	1000	180	0.6530
3	1050	180	0.4887
4	1100	180	0.3012
5	1150	180	0
6	1200	180	0
7	1250	180	0.5330

Table (10) Weight loss in 10% H₂SO₄ at 200 °C

NO.	Sintering Temp. (°C)	Time (min)	Weight Loss (%)
1	950	180	0.7446
2	1000	180	0.5263
3	1050	180	0.2232
4	1100	180	0
5	1150	180	0
6	1200	180	0
7	1250	180	0.4287

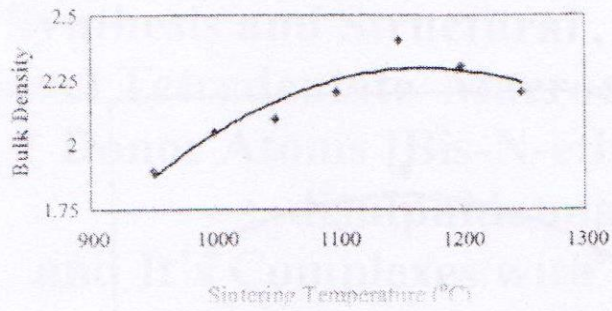


Fig (1) Effect of sintering temperature on bulk density of specimen

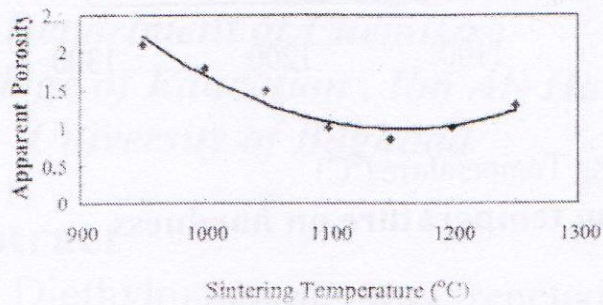


Fig (2) Effect of sintering temperature on apparent of specimen

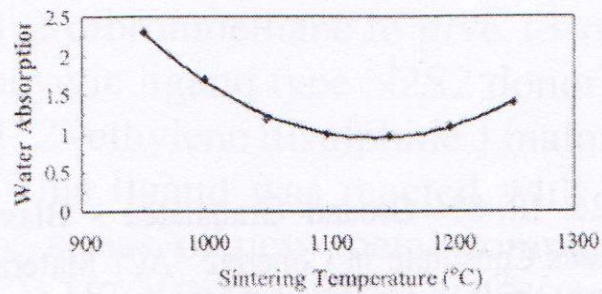


Fig (3) Effect of sintering temperature on water absorption

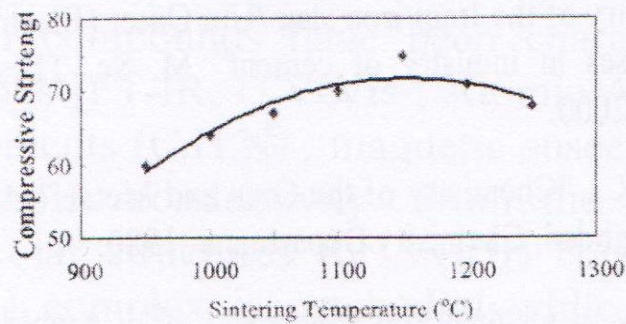


Fig (4) Effect of sintering temperature on compressive strength

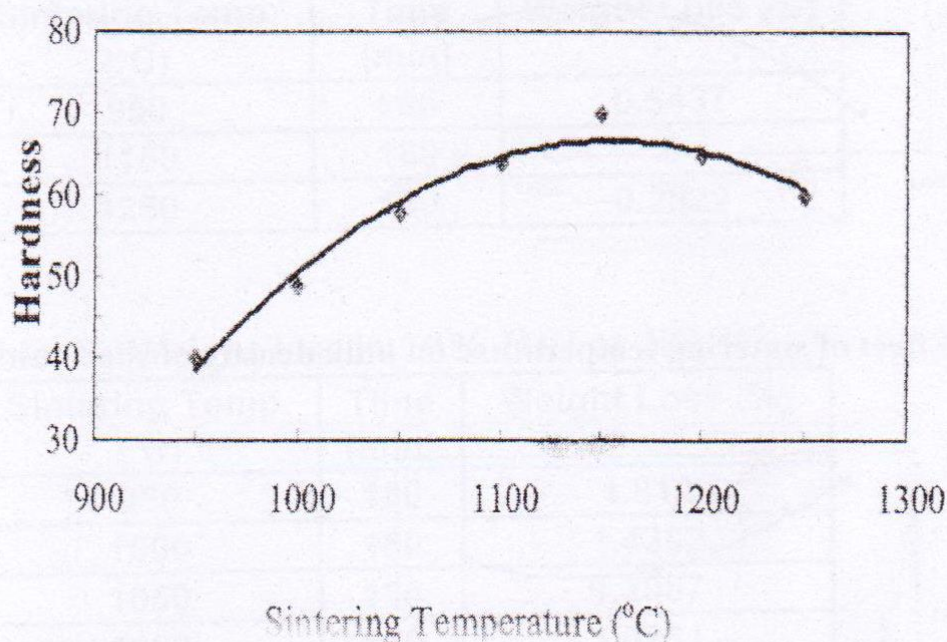


Fig (5) Effect of sintering temperature on hardness

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