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PRODUCTION OF CERAMICS FROM WASTE GLASS AND JORDANIAN OIL SHALE ASH

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Abstract. *In this study, a* 2^3 *full factorial design methodology was applied to study the effect of three process input variables, namely ash content (30 and 50 wt%), pressing pressure (120 and 150 MPa) and heat treatment temperature (900 and 1050 °C) on the characteristics of ceramics. The ceramics specimens were prepared from oil shale ash and waste glass by pressing followed by heat treatment. The oil shale was obtained from El-Lajjun region in Jordan. Three response variables were chosen to investigate the process variables of interest. The response variables were water absorption, chemical absorption and bending strength.*

It was found that higher heat treatment temperatures are favored to obtain ceramics with lower water absorption, lower chemical absorption and higher bending strength. The main effects of ash content and pressing pressure on the characteristics of ceramics were found to be shared by interaction effects. The factorial design methodology revealed the interaction and its effect among process input variables on the characteristics of ceramics.

Ceramics with minimum water absorption (7.7%), minimum chemical absorption (9%) and bending strength (41 MPa) were obtained based on the following optimized conditions: 30 wt% ash content, 120 MPa pressing pressure and 1050 °C heat treatment temperature.

Keywords*: oil shale, ash, waste glass, ceramics, factorial design.*

1. Introduction

The beneficiation of waste is a necessity in the world of increasing population and with inherent and strict environmental constraints. Waste can be generated from industrial, commercial, mining and agricultural operations and from community activities. Industrial waste left over from mining and manufacturing processes might hinder the economic feasibility of such processes due to the continuous need for maintaining waste disposal facilities. Waste transformation into valuable resources enhances sustain-

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ability and reduces pollution. Ash is an example of industrial waste which has received considerable attention in the last few decades. Ash is a waste produced from the incineration or combustion of a wide spectrum of materials, such as wood, coal and oil shale. It is estimated that more than 650 million tons of coal ash is produced annually [1].

Jordan is one of the top leading countries in terms of oil shale resources. About 50 billion tons of oil shale deposits is available in Jordan [2]. It is expected that Jordan will utilize the oil shale for energy production to reduce its dependency on fossil fuel. Recently, the Government of Jordan has signed a memo of understanding for a partnership with local and international partners to build an oil shale-fired power plant to produce electricity and another one for shale oil production [3]. The two plants will utilize 10 million tons of oil shale per year to produce 554 MW of energy. Based on the proposed plant capacity, it is expected that 5 million tons of ash will be generated yearly due to the fact that Jordanian oil shale contains almost 50% ash [4]. It has been proposed that the ash will be handled by dumping. However, thinking of utilizing oil shale ash as a resource more than a waste will support the national economy and reduce pollution.

The utilization of oil shale ash has been investigated for many engineering applications. Studies have shown that oil shale ash can be utilized in the stabilization of problematic soils [5], for Portland cement production [6], for zeolite production [7], for cement-treated base production [5], as a concrete binder [8, 9], as a modifier for asphalt binders [10], for self-compacting concrete production [11], for asphalt-mix production [12], for ceramics production [13–15], for wastewater treatment as an adsorbent material [16, 17] and for gas purification [18].

In this study, the oil shale ash is investigated toward its utilization for ceramics production. Waste glass will be incorporated with the oil shale ash to produce ceramic material. The effect of ash weight fraction, pressing pressure, and heat treatment temperature on the physical and chemical properties of ceramics is investigated by applying a 2^3 full factorial design methodology. The main objectives of this study are to identify the most influential process operating conditions on the production of ceramics and the effect of interaction among process variables on the physical and chemical properties of the produced ceramics.

2. Experimental

2.1. Preparation of oil shale ash

Oil shale samples were collected from El-Lajjun region located 100 km south of Amman, Jordan. The samples were collected from different positions of the deposit. The samples were mixed together, crushed and then screened. In this study, a size fraction of 70–125 μm was used. Typical ashing procedures were followed for the preparation of oil shale ash [2, 4, 18]. The samples of oil shale were placed in a ceramic crucible, and then heated in an electrical furnace to a temperature of 900 °C at a rate of 10 °C/min. The samples were held in the furnace at a specified temperature for 6 h to guarantee complete removal of water and organic matter. After that, the furnace was switched off, and the samples were cooled down to room temperature inside the furnace. Typical properties of El-lajjun oil shale ash are presented in Table 1 as determined by X-Ray Fluorescence Spectrometry (XRF) [9].

Table 1. Chemical composition of El-Lajjun oil shale ash [9]

Constituent	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O
Wt%	19.6	6.0	2.2	55.24	0.9	8.5	0.56	0.3

2.2. Preparation of waste glass powder

Waste glass bottles were collected, carefully washed and dried in an oven at 60 °C for 24 h. The glass bottles were crushed, ground in a disk type ball mill, and then screened. In this study, the used waste glass size fraction was < 75 μm.

2.3. Preparation of ceramic samples

Different powder mixtures (100 g each) were prepared by mixing the waste glass powder with the oil shale ash at different proportions in a conical mixer (PASCALL, LAB Mixer II) for 25 minutes. No binder was used in the mixture. After mixing, each mixture was pressed under different loads into a cylindrical shape having a diameter of 30 mm and a length of 8 mm.

After pressing, the samples were subjected to heat treatment. The prepared samples were heated up to a specified temperature at a rate of 10 °C /min in an electrical furnace, then held at a specified temperature for 4 h. After that, the furnace was switched off, and the samples were cooled down to room temperature inside the furnace. Two heat treatment temperatures were investigated in this work, namely 900 and 1050 °C. The selected heat treatment temperatures are based on typical operating conditions reported in literature for similar systems [19–21]. Heat treatment at these temperatures ensures the formation of crystalline phase in the glass ceramic. The duration of thermal treatment was fixed at 4 h to ensure sintering and complete crystalline phase transformation.

2.4. Characterization of samples

Sample characterization was carried out by examining water absorption, the bending strength and chemical absorption.

The water absorption was determined from the difference between dry mass and surface dry mass after immersion in water at a temperature of 20 °C for 8 h. The determination of the bending strength of the prepared samples was carried out by subjecting the samples to a 3-point bending strength test. The chemical absorption test was carried out by the measurement of the weight difference between dry mass and surface dry mass after immersion in sodium hypochlorite solution (5.5%) at 20 °C for 4 h.

3. Experimental factorial design and analysis

In the experimentation for ceramics production from waste glass and oil shale ash it is necessary to consider the most influential factors that might affect the quality of the produced ceramics. To achieve this goal, a full factorial design methodology was followed to identify the main effects of three processing factors on the quality of the produced ceramics. The three factors studied were percentage of ash used (X_1), pressing pressure (X_2) and heat treatment temperature (X_3). Each of the three factors was studied at two levels (Table 2). Therefore, the arrangement and number of experiments is considered to be a 2 × 2 × 2 or 2^3 factorial design. The eight formulations are shown in Table 3 with variable levels coded with plus and minus signs. In addition, the full factorial design methodology enables studying the interactions among the selected factors. The prepared ceramic samples were subjected to the following tests (response output variables): water absorption (Y_1), chemical absorption (Y_2), and bending strength (Y_3).

Yates's algorithm was followed for the determination of main and interaction effects [22].

Table 2. Process input variables and their levels

Input variable	Ash content, wt% X_1		Pressing pressure, MPa X_2		Temperature, °C X_3	
Level	Low (–)	High (+)	Low (–)	High (+)	Low (–)	High (+)
Condition	30	50	120	160	900	1050

Table 3. Experimental design matrix and response of output variables

Run	Input variables			Output variables		
	X_1	X_2	X_3	Y_1, %	Y_2, %	Y_3, MPa
1	–	–	–	10.8	13.7	10
2	+	–	–	18.1	20.6	7
3	–	+	–	11.4	12.8	13
4	+	+	–	17.9	12	15
5	–	–	+	7.7	9.3	41
6	+	–	+	10.2	14.5	18
7	–	+	+	11.1	23	21
8	+	+	+	7.6	6	10

4. Results and discussion

4.1. Main and interaction effects of process input variables on the water absorption of ceramics

The main effects of process variables on the characteristics of ceramics have been studied. Table 4 shows the calculated main effects based on all experimental observations. Figure 1 depicts the main effect plots for the studied process variables.

Table 4. Full 23 factorial design analysis of process response

Term	Response		
	Y_1	Y_1	Y_3
X_1	3.2	–1.4	–4.2
X_2	0.3	–1.1	0.4
X_3	–5.4	–1.6	15.6
X_1X_2	–1.7	–7.5	8.8
X_1X_3	–3.7	–4.5	–3.9
X_2X_3	0.1	–3.7	–5.5
$X_1X_2X_3$	–1.3	–3.6	6.5

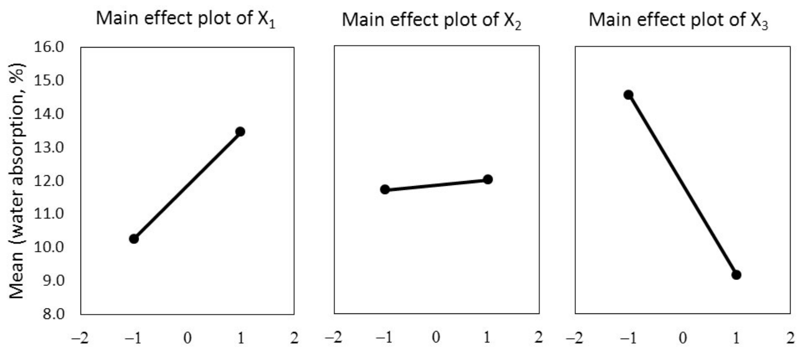

Fig. 1. Plots of the main effect of process input variables on water absorption.

It can be seen that the heat treatment temperature and ash content have significant effects on water absorption. However, and within the range of experimental conditions, the pressing pressure has a little effect on changing the water absorption. The absolute magnitude of effects of both heat treatment temperature and ash content are one order of magnitude higher than the absolute magnitude of the effect of pressing pressure. Increasing the ash content from 30 to 50 wt% increased slightly water absorption from 10.3 to 13.5%. Increasing the heat treatment temperature from 900 to 1050 °C reduced water absorption from 14.6 to 9.2%. Increasing the pressing pressure from 120 to 150 kept water absorption rate at a constant value of 11.8%. High temperature treatment of ceramics reduced the capacity of ceramics toward water absorption. This might be attributed to the sintering effect and reduction in porosity. Sintering at high temperature enhanced the bonding and compacting between the powder constituents. Investigations by other researchers indicated that increasing the heat treatment temperature of glass ceramics made of sludge bottom ash and waste glass decreased the pores in the matrix, leading to a high density of glass ceramics [19].

Each effect of process variables on water absorption must be carefully examined at different levels of the other process input variables. This can be achieved by constructing the plots of interaction between process input variables (Fig. 2).

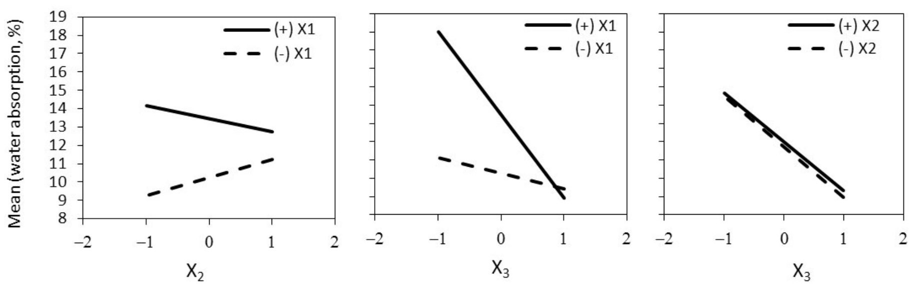

Fig. 2. Plots of the interaction effect of process input variables on water absorption.

Figure 2 shows that there is a moderate yet important interaction between ash content and pressing pressure. Increasing the pressing pressure at lower ash content increased the water absorption from 9.3 to 11.3%. On the contrary, increasing the pressing pressure at higher ash content decreased the water absorption from 14.2 to 12.8%. The ash used in this study was less than 75 µm in size with a predominant portion as fine powder. Therefore, samples with high ash content could be readily pressed. This led to enhancement in the bonding and compacting between the powder constituents, thereby, reducing the water absorption.

In addition, another strong interaction exists between ash content and heat treatment temperature. Increasing the heat treatment temperature at lower ash content fairly decreased the water absorption from 11.1 to 9.4%. However, increasing the heat treatment temperature at higher ash content sharply decreased the water absorption from 18 to 8.9%. Higher ash content means higher calcium oxide content. This led to a strong interaction with the glass silica at higher temperature, thus, forming a crystalline phase and a microstructure with better physical properties in terms of water absorption.

On the other hand, there is a negligible interaction between pressing pressure and heat treatment temperature. Increasing the heat treatment temperature for both levels of pressing pressure decreased the water absorption at almost the same magnitude from 14.5 to 9.1%.

Ceramic tiles are classified by the ISO 13006 technical standard on the basis of water absorption as follows: < 3% (Group I), 3–10% (Group II) and > 10% (Group III) [23]. In this study, the water absorption of ceramics varied from 8.9 to 18 wt%. Therefore, they are classified as group III ceramics. Ceramics with this classification are best suited to be employed as wall tile ceramics.

4.2. Main and interaction effects of process input variables on chemical absorption

Table 4 shows the calculated main effects of process input variables on chemical absorption. Figure 3 depicts the plots of the main effect of the studied process input variables on chemical absorption.

The absolute magnitude of effects of all process input variables are close to each other and have the same trend, that is, increasing the ash content, heat treatment temperature, or pressing pressure tends to barely decrease the chemical absorption. However, a strong interaction exists among the process input variables. Figure 4 shows the plots of the interaction effect of process input variables on chemical absorption.

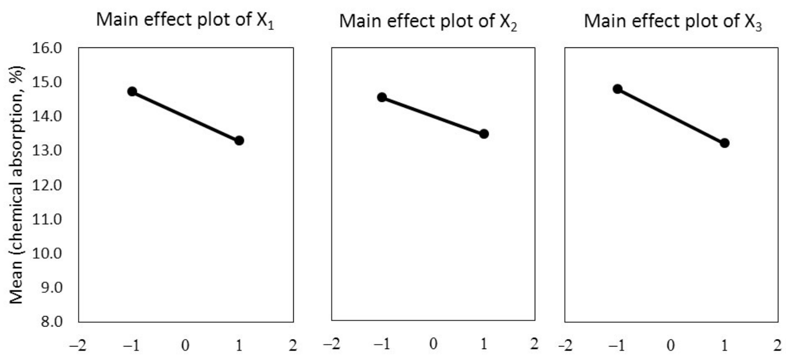

Fig. 3. Plots of the main effect of process input variables on chemical agent absorption.

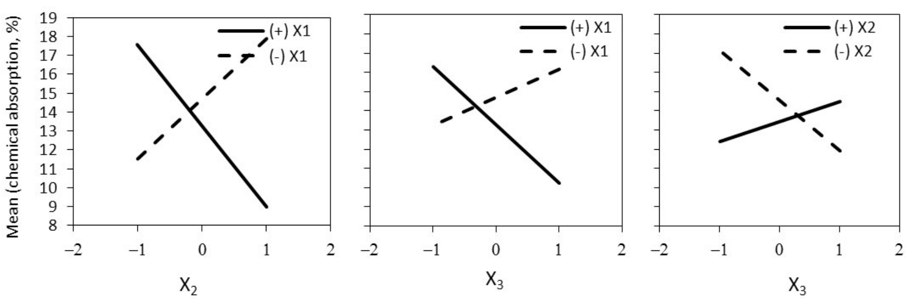

Fig. 4. Plots of the interaction effect of process input variables on chemical agent absorption.

The interactions among the process input variables are of antagonistic type [24]. Minimum chemical absorption was observed when the ash content and pressing pressure were kept at high level. Furthermore, increasing the heat treatment temperature enhanced the chemical durability by reducing the chemical agent absorption.

4.3. Main and interaction effects of process input variables on bending strength

Table 3 shows the calculated main effects of process input variables on the bending strength. Figure 5 depicts the plots of the main effect of the studied process input variables on the bending strength.

The mechanical properties of the prepared ceramics samples in terms of the bending strength are highly affected by the heat treatment temperature. The relative magnitude of the effect of the heat treatment temperature on the bending strength was four times higher than the main effect of ash content and one order of magnitude higher than that of pressing pressure. The main effect plot for the heat treatment temperature shows that increasing the heat treatment temperature from 900 to 1050 °C increased the bending strength from 11 to 27 MPa. This might be due to the densification caused by heat treatment. Increasing the heat treatment temperature increased the density of the sample by reducing its porosity and eventually increasing its bending strength. The trend in results is in agreement with other investigations on similar systems. It has been reported that mechanical properties of clay structural ceramics containing coal fly ash were highly dependent on the processing conditions. The results of the investigation indicated that the density of the prepared ceramics increased with increasing firing temperature due to reduction in porosity [25]. In another investigation concerning the production of ceramics from coal fly ash, it was found that increasing the heat treatment temperature generally increased the bending strength as a result of the change of morphology of the fly ash particles. Consequently high density/ lower porosity compacts were obtained at high treatment temperature [26].

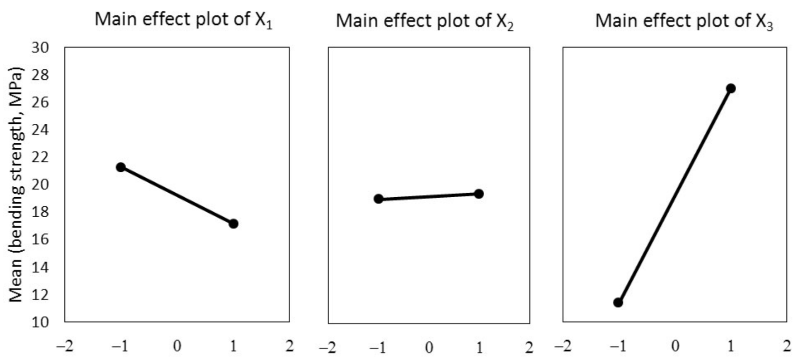

Fig. 5. Plots of the main effect of process input variables on bending strength.

Ash content had a noticeable main effect on the bending strength of the prepared ceramics. Increasing the ash content from low level settings to high level settings decreased the bending strength from 21 to 17 MPa. The high ash content is counterbalanced by a lesser amount of waste glass; this in turn will reduce the glassy phase responsible for filling the pores in the ceramic matrix. As a result, reduction in bending strength was noticed. However, this effect must be connected with the possible effect of interaction between the other process input variables. This can be confirmed by constructing the interaction effect plots. Figure 6 shows the plots of the interaction effect of process input variables on the bending strength.

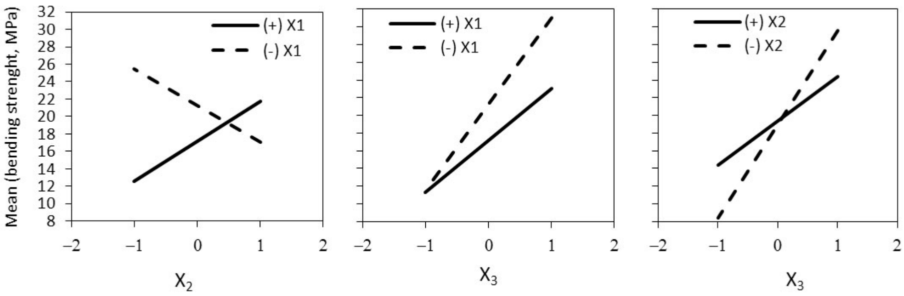

Fig. 6. Plots of the interaction effect of process input variables on bending strength.

The results indicate a strong interaction to exist between ash content and pressing pressure. Increasing the pressing pressure at higher ash content settings increased the bending strength from 13 to 22 MPa. However, increasing the pressing pressure at lower ash content settings decreased the bending strength from 25 to 17 MPa. This is in agreement with the earlier explanation that higher ash content could be readily pressed, leading to a reduction in the porosity of ceramics. The lower porosity ceramics obtained under higher ash content and higher pressing pressure possessed high bending strength values.

In addition, Figure 6 shows that the interaction between pressing pressure and heat treatment pressure is of antagonistic type. Enhancement in the bending strength could be achieved under high temperature treatment and low pressing pressure. Otherwise, a high pressing pressure must be applied if low temperature treatment is to be employed.

On the other hand, there is a negligible interaction between ash content and heat treatment temperature. Increasing the heat treatment temperature from 900 to 1050 °C increased substantially the bending strength for both high and low ash content ceramics.

Within the operating conditions applied in this study, the bending strength of the prepared ceramics ranged from 7 to 41 MPa. Considering that for group III ceramics with water absorption more than 10 wt%, a bending strength value of 15 MPa is prescribed by the standard (EN14411/ISO 10545-4) [27], it is observed that the ceramics produced in this study may exhibit satisfactory mechanical strength if proper process input variables are applied. For example, at lower ash content (30 wt%), lower pressing pressure (120 MPa) and high temperature treatment (1050 °C), ceramics with a 41 MPa bending strength can be produced, which can be used for a wide range of applications.

5. Conclusions

Ceramics was successfully prepared from oil shale ash and waste glass by using the mechanical milling method. A 2^3 full factorial design analysis was effectively performed to assess the effect of ash content, pressing pressure and heat treatment temperature on the mechanical and chemical properties of glass ceramics.

Water absorption was highly affected by ash content and heat treatment. Increasing the heat treatment temperature at lower ash content fairly decreased the water absorption However, increasing the heat treatment temperature at higher ash content sharply decreased the water absorption.

Chemical absorption was affected by ash content and pressing pressure. In addition, high temperature treatment assisted in reducing chemical absorption.

The bending strength of glass ceramics was substantially enhanced by increasing the heat treatment temperature. Increasing the pressing pressure at higher ash content increased the bending strength. However, increasing the pressing pressure at lower ash content decreased the bending strength.

The chemical and mechanical properties of ceramics prepared in this study are good enough for the ceramics to be utilized for various purposes such as building materials and engineering applications.

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