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# Development and Characterization of Ultra Low Cement Castable Cordierites by Thixotropic Properties Mixtures

**Ana M. Paniagua-Mercado[1], Arturo Méndez-Sánchez[1], Elvia Díaz Valdés[1], Concepción Mejía García[1], Paulino Estrada Díaz[2]**
[1]Depto. de Física, Escuela Superior de Física y Matemáticas, Instituto Politécnico Nacional, Col. Lindavista, México
[2]Sector-Ceramics and Refractories, Manuchar International, México
Email: ampani@esfm.ipn.mx, pestrada@manuchar.com.mx

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## ABSTRACT

The main target in this investigation was to take advantage of the reology properties of the tixotropic mixes in Ultra Low Cement Castables (ULCC). The cordierite phase in refractory mix can be obtained using raw materials with magnesium oxide in its composition, such as, $Mg(OH)_2$ or $H_2Mg_3(SiO_3)_4$ (Talc mineral), with a content of 63.5% $SiO_2$, 31.7% MgO and 4.8% $H_2O$. In this investigation, as magnesium source, a commercial calcined magnesite with 90% MgO was used. This mineral was selected instead of Talc mineral, because this last contains more impurities in its composition that tend to form more amounts of liquid phases with low fusion points. For this work two different ULCC mixes were designed. These were fired at 1260ºC, the cordierite phase was quantified in each mix.

**Keywords:** Reology; Thixotropic Mixtures; Ultra Low Cement Castables; Cordierite

## 1. Introduction

There are in the market, several industrial methods and a wide of conventional raw materials to produce cordierite mixes ($MgO \cdot Al_2O_3 \cdot SiO_2$). The complete phase transformation is around 1300ºC depending on the raw material used in the mix [1]. The industrial application is mainly focus where high thermal shock resistant, low thermal expansion and corrosion resistance are demanded. Such is the case of the cordierite refractory plates used for the conventional firing of sanitary and tableware, or recently used as substrate material in microelectronics [2]. The main manufacturing processes to develop cordierite phase are the sol-Gel method [3], co-precipitation [4], solid-state reaction [5] and by slurry. Aluminosilicate based ultra low cement castables (ULCC) are widely used mainly in the Steel and cement industries due to improved refractory properties at high temperatures. The bonding system in ultra low cement castables is achieved by using high alumina calcium aluminate cement.

Increasing the cement content in the concrete mix also increases the amount of liquid phases as the anorthita ($CaAl_2Si_2O_8$) and gelenite ($Ca_2Al_2SiO_7$). These both tend to reduce the amount of free silica and decrease, in an important way, its chemical corrosion resistance. This last affects negatively their mechanical properties at high temperatures and in consequence the thermal shock resistant comes down. The lime/silica ratio is very important in the formation of liquid phases and its viscosity at high temperatures, because it affects the strength and corrosion resistance [6]. The use of very low amounts of high alumina cement in ULCC is principally to avoid the liquid phases formation inside the refractory matrix. Other important variable is the particle size distribution because it has a major impact in the reology of ULCC and in the final physical properties [7].

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## 2. Experimental Procedure

**Table 1** shows the chemical formulation of the two concrete mixes tested in this investigation. For each one of the designed mixes the preparation was as follows. First, the raw materials were dry mixed for at least 1 minute, after that, the deflocculant (sodium tripolifosfate); the polypropylene fibers and the high alumina cement were added to the final mix. At once, the water was added slowly and mixed 10 second more. After the mix was placed in a vibratory table (3000 cps), for no more than 30 seconds up to the mix, it got a thixotropic behavior.

After the vibratory step, the mix was allowed to dry and it was set for 12 hours at room temperature (25ºC). Afterwards the mix was dried 24 h in a laboratory stove at 110ºC. Finally, the mix was calcined between 1260°C - 1280°C during 5 h, in a gas furnace. In order to determine the cordierite forming and main phases present, the calcined samples were analyzed by X-ray diffraction in a Siemens D-500 diffractometer, with Kα of Cu in the Bragg-Brentano configuration, in a 2 range of 10º-120º. Micrographs of each one of the two calcined mixes were obtained with secondary electrons, at 800X of magnification, in a scanning electron microscopy JEOL-6300.

**Table 1. Chemical Formulation of Mixtures.**

| Compound | MIX I (%) | MIX II (%) |
|---|---|---|
| $Al_2O_3$ | 47.92 | 47.49 |
| $SiO_2$ | 20.86 | 41.02 |
| CaO | 0.80 | 1.08 |
| MgO | 27.15 | 7.37 |
| $Fe_2O_3$ | 1.08 | 0.82 |
| TiO | 1.22 | 1.19 |
| $K_2O$ | 0.65 | 0.66 |
| $Na_2O$ | 0.29 | 0.31 |

## 3. Results and Discussion

### 3.1. Chemical Analysis

Specific raw materials were selected to design the ULCC cordierites. Oxide or other non-clay powders generally have poor workability when they are mixed with water and settling rapidly at lower water levels. For this reason, it was utilized some deflocculating chemicals to get a thixotropic behavior. In ULCC mix the amount of water is a problem because higher amount of water, than that required, is to the detriment of the final physical properties of the refractory concrete as %Porosity and density (see **Table 2**).

### 3.2. Physical Properties

The physical properties of the mixtures were calculated and they are shown in **Table 3**.

The physical properties reported for the mix I, with a greater amount of fine particles than for mix II, table III, the fines are because the raw materials used for mix I are more than for mix II, show that it was required more water in its preparation than for mix II.

It can be also observed from mix I, that at higher water content in the mixII, the density decreases and the porosity too, **Table 3**.

Increases water resulting in a negative effect for the final physical properties. For Mix II, with a greater amount of coarse particles, until the raw mix, the demand of water was lower than for mix I, and this was reflected in its final physical properties.

### 3.3. X-ray Diffraction

In the patterns of X-ray diffraction shown in **Figure 1**, the different phases contained in the mixes I and II are presented. These were quantified with the peaks getting by X-ray diffraction of mixes burned.

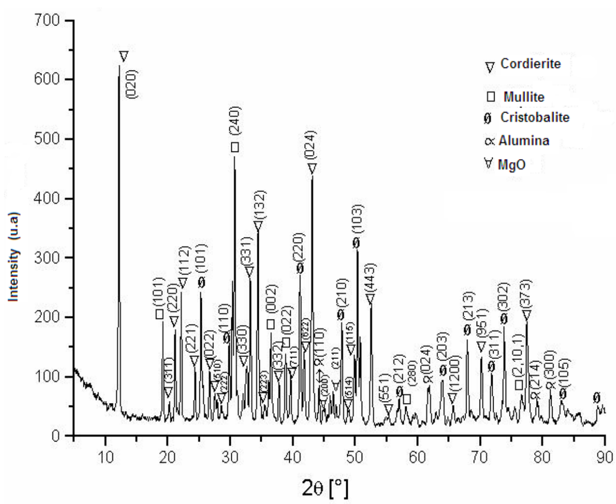
**Table 2. Physical properties of the mixtures.**

| Mix/ Property | Setting Time (min) | Water Mixed (mL) | Density ($g/cm^3$) | Absorption (%) | Porous (%) |
|---|---|---|---|---|---|
| MIX *I* | 160 | 12.5 | 1.95 | 13.8 | 27.0 |
| *MIX II* | 80 | 8.5 | 2.07 | 9.6 | 20.0 |

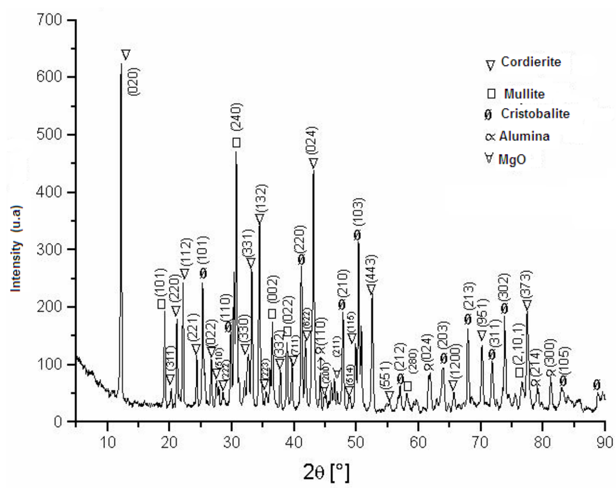
**Table 3. Granulometry and density of raw materials.**

| Raw materials | Particle Size (μm) | Density (g/ml) |
|---|---|---|
| Magnesium Oxide | 590 | 3.10 |
| Calcined Kaolin | 74-2380 | 2.52 |
| Microsilica | 44 | 2.70 |
| High Alumina Cement | 53 | 0.90 |
| Calcined Bauxite | 74 | 3.15 |

The X-ray diffraction patterns reported a higher amount of cordierite phase for mix I than for mix II, as it is observed in **Table 4**. The mix I had a big amount of fine particles which contributed in an important way to the formation of the cordierite phase, however, it was utilized a bigger amount of water for its preparation and the physical properties for this mix were poor in comparison with those ones of the mix II. Other main phases detected in this analysis were mullite, cristobalite, alpha-alumina and magnesium oxide. It is necessary to remark that particulate size distribution is a very important factor in the quantity and type of phases formed for this ULCC.




(a)



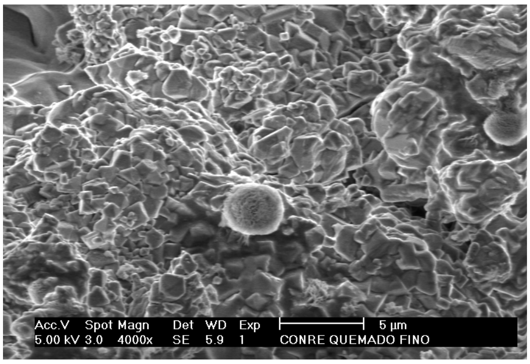

(b)

**Figure 1. Patterns of X-ray Diffractions: (a) mix I, (b) mix II burned.**

**Table 4. Phase quantity in the mixtures.**

| MIX/ Phase | Cordierite (%) | Mullite (%) | Cristobalite (%) | α $Al_2O_3$ (%) | MgO (%) |
|---|---|---|---|---|---|
| ***MIX I*** | 31.87 | 18.64 | 35.23 | 8.04 | 6.22 |
| ***MIX II*** | 14.27 | 29.00 | 37.29 | 5.18 | 14.26 |

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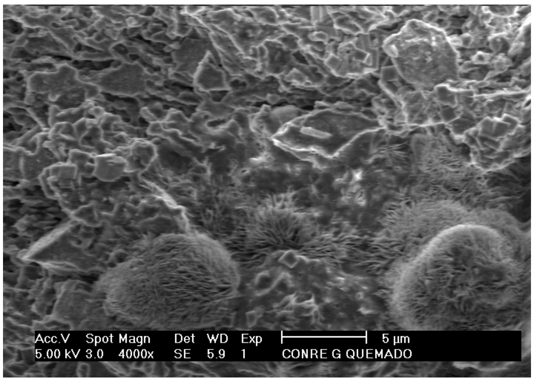


(b)

**Figure 2. Micrographs: (a) mix I and (b) mix II.**

### 3.4. Scanning Electron Microscopy

In the micrographs shown in **Figure 2** we can observe material like flakes corresponding to cordierite, and liquid phases with composition of $Fe_2O_3$, $Na_2O$, $K_2O$ and GeO formed with the presence of $SiO_2$ and detected by Micro analyses of SEM, all of these have low fusion points.

## 4. Conclusions

1. It was processed a refractory Cordierite ULCC mix, with level of cordierite phase commercially acceptable and with thixotropic properties.
2. The main physical properties of this type of ULCC are subjected to a very restricted particle sizes distribution to obtain the better physical properties after fired.
3. Further research work must be doing to improve in a better way the cordierite performance phase and also the final physical properties of the castable.

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