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Investigation of novel waste glass and limestone binders using statistical methods

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HIGHLIGHTS

- Novel binders based on urban waste glass and limestone were studied.
- The Taguchi method is useful for the optimization of the experimental work.
- Statistical analysis allowed to predict and maximize the strength.
- The hydration products were amorphous C–S–H, silica gel, pirssonite and gaylussite.
- The incorporation of Na_2CO_3 caused a synergistic effect that increased the strength.

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ABSTRACT

This paper presents a study on new alternative binders based on waste glass and limestone activated by NaOH and Na_2CO_3. The Taguchi method was applied, to optimize the experimental work while keeping statistical significance, considering the factors: CaO/SiO_2 ratio, % Na_2O and curing temperature; their effect was analyzed on the compressive strength using signal to noise ratios. The optimal conditions included $CaO/SiO_2 = 0.5$, activated at 40 °C with 9% Na_2O using Na_2CO_3/NaOH; the strength of the confirmation experiments agreed with the predicted values. The reaction products were: calcium silicate hydrate, a silica gel type phase, and crystalline phases like pirssonite and gaylussite.

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1. Introduction

1.1. Glass and limestone as raw materials

The production of the most common binder, Portland cement (PC), emits large amounts of CO_2. In 2013 the world production of PC reached 3500 million tons [1], which meant more than 490 Kg per capita and that the cement industry was the source of about 10% of CO_2 emissions worldwide [2]. Green alternative binders are of interest to reduce this environmental impact. Some alternatives based on natural raw materials or industrial byproducts have been explored regarding their potential as binder for construction [3–5]. Blast furnace slag and fly ash are among the most studied as partial or total replacements for PC in concrete due to their wide availability [6,7]. Nonetheless, a variety of other wastes or natural raw materials with cementing potential are available and their use in concrete becomes increasingly attractive if there are also environmental issues related to their disposal; such is the case of waste glass [8].

Glasses of the family SiO_2–CaO–Na_2O take a major share of the total solid wastes disposed worldwide. As glass is not biodegradable, landfilling does not provide the most environmentally friendly solution; so recycling turns out a better alternative [9]. Urban Waste Glass (UWG) is a promising alternative cementitious material that require relatively minor processing infrastructure to make it useful. A number of studies have examined the use of UWG in cement and concrete [10], such as a raw material in the manufacture of PC, as an aggregate, inert filler or as partial PC replacement [9]. Oliveira et al. [11] reported that mortars with 10–20% incorporation of fine glass as filler, increased the flexural strength and compressive strength by 29–86% and 31–91% respectively, relative to the conventional mortar. Schwarz and Neithalath [8] studied the influence of fine glass powder as replacement of PC, they noted that the use of 5–20% of glass did not accelerate the setting or increase the early age strength of cement pastes; however, the glass powder exhibited a pozzolanicity similar or greater than that of fly ash.

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Additionally, silicoaluminate and silicate glassy materials, such as UWG, are susceptible to activation by alkaline compounds and depending of the availability of calcium, can form silica gel or calcium silicate hydrates [12–14]. Redden et al. [7] reported that for a glass powder binder activated with 4 and 8 M NaOH, the main reaction product was a sodium silicate gel. Alkali activated UWG could be mixed with other supplementary cementitious materials to form stable binders [15–17]. In order to compensate the chemical deficiency of glass in CaO and to obtain stable reaction products such as C–S–H, in this investigation the limestone was chosen as a complementary raw material.

Limestone (LS) is a low price abundant sedimentary rock that represents an interesting alternative raw material in alternative binders [18]. Several studies have shown the beneficial effects of limestone additions on both the fresh and hardened properties of various binary and ternary cementitious systems based on PC [19–24]; these effects explain why regulations worldwide permit its addition as filler or replacement to the latter [19,25]. Reports on the use of limestone on alkali activated binders are not abundant. Yip et al. [19] studied the effects of calcite and dolomite on the strength of metakaolin-based geopolymers and noted that the addition of about 20% calcite or dolomite improved the strength, also, that more than 20% had a deleterious effect due to a disruption of the geopolymer gel network and reduced reactive aluminosilicate content. Such authors did not observe distinct calcium silicate hydrate phase formation, although some dissolution of the mineral particles was observed, but with no major effect in most instances; the mineral particles interacted with the geopolymer gel predominantly via surface binding, which appeared to be somewhat stronger in the case of calcite than dolomite. A report by Yin et al. [26] on binders of 100% of three $CaCO_3$ rocks activated with waterglass, the 28 day strength were 10.3 and 40.48 MPa, for contents of 0.7 and 19.7% MgO, respectively; nonetheless, the authors did not present information about the amount of Na_2O used or the modulus of waterglass used. A study of mixtures of carbonatite with blast furnace slag activated with sodium silicate [27] noted that strength increased with the slag content and concentration of sodium silicate, but higher modulus of the latter had a negative effect; they pointed that the corrosion of the carbonatite in the alkaline environment released Ca and Mg, reduced the particle size and improved packing and strength, but they did not comment on the reaction products formed.

1.2. Taguchi method

A technique for defining and investigating all possible conditions in an experiment covering or encompassing multiple factors, is known as a factorial design of experiments. The purpose of any experimental design is to obtain information of quality for understanding, improve, test hypotheses and make relevant decisions on a research or industrial problems [28]. In 1957, G. Taguchi proposed the use of orthogonal arrays, which significantly reduces the number of experiments; later he complemented his method leading to the term Taguchi method in 1980 [29,30].

The aim of a parameter design of experiments is to identify and design the setting of the process parameters that optimize the chosen quality characteristic, which are least sensitive to noise factors [31]. The Taguchi method uses the signal to noise ratio (S/N), which is recommended for multiple testing, since it allows to determine the optimal conditions of the experimental design based on the characteristic of quality desired among three categories: larger the better, smaller the better and the nominal the better. In order to determine the reliability of the experimental results and the degree of the effect of the factors on the results, the analysis of variance (ANOVA) is used with a given confidence level.

This papers reports the results from an investigation on the strength, reaction products and microstructures of on alkali activated cementitious matrices based on urban waste glass and limestone, analyzing the effect of several factors. The Taguchi method was used to optimize the experimental work while keeping the statistical significance of the work, with the advantages of reducing the influence of external noise sources and determining optimum working conditions from laboratory work that can be reproduced in the field [28].

2. Experimental procedure

2.1. Raw materials

Urban waste glass (UWG) compounded by a mixture of amber glasses and clear glasses was used. The UWG was washed, dried, crushed and ball milled until passing the #325 sieve. A local commercially available limestone sand was used after milling to pass the #325 sieve. The chemical composition of both raw materials is given in Table 1.

2.2. Dissolution of limestone and urban waste glass in alkaline solutions

A set of experiments was established to analyze the dissolution of UWG and LS under alkaline environments, Table 2 describes the five tests carried out, which included the separate LS and UWG and a composite indicated as optimal by the Taguchi method. For all tests, 5 g of LS–UWG were diluted in 100 ml of solution with 9% Na_2O relative to the weight of the powders; the mixture was left under constant agitation for 24 h at 40 °C. The final solution was filtered and stored, in sealed plastic bottles, for characterization by inductively coupled plasma atomic emission spectroscopy (OPTIMA 8300, Perkin–Elmer).

2.3. Design of experiments

The selection of the seven factors and their levels, indicated in Table 3, was made on the basis of previous experiences with various alkali activated binders and on preliminary trial experiments; these are described as follows. The CaO/SiO_2 weight ratio, defined based on the proportion and composition of the UWG and LS used in the binders. A pre activation (PA) process was introduced and considered within two of the factors: % with-PA, that indicates the %wt. of UWG, LS or their mixtures that was pre mixed (pre activated) with the whole of the alkaline solution before adding the remaining dry powdered ingredients; on the other hand, PA-order indicates which of the UWG, LS or their mixtures, was first added to the alkaline solution. The other factors were the type of activator and the amount (% Na_2O) used relative to the weight of UWG + LS, water/binder ratio (W/B), and curing temperature.

Table 2
Experiments established to analyze the dissolution of raw materials.

Dissolution tests	Raw material (wt%)		Alkaline activator
	LS	UWG	
D1	100	–	NaOH
D2	100	–	Na_2CO_3
D3	–	100	NaOH
D4	–	100	Na_2CO_3
D5	30	70	Na_2CO_3/NaOH

Table 1
Chemical composition (%wt.) of waste glass and limestone by X-ray Fluorescence.

Compound	SiO_2	CaO	$CaCO_3$	Na_2O	Al_2O_3	MgO	K_2O	Fe_2O_3
Glass (UWG)	70.34	12.76	–	13.58	1.52	0.35	0.525	0.397
Limestone (LS)	1.44	–	97.29	0.132	0.318	0.457	0.106	0.124

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