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# Synthesis of a Geopolymer Binders Using Spent Fluid Catalytic Cracking (FCC) Catalyst

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**Abstract.** A synthesis of a geopolymer binder using spent fluid catalytic cracking (FCC) catalyst is evaluated in this research. Spent FCC catalyst is a solid waste product from the oil refining industry worldwide. It is composed mainly by aluminium and silicon oxides. The silica modulus (Ms), NaOH molar concentration, and liquid/solid (AS/FCC) weight ratio were studied as key factors of the mixture composition for the evaluation of the compressive strength and bulk density of geopolymers. Additional total ratios of SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>O/SiO<sub>2</sub>, and H<sub>2</sub>O/total solids, which consider chemical composition of the final mixture, were studied. Results indicate that there is an optimum value for each Ms, NaOH molar concentration and AS/FCC that produces the maximum compressive strength. These values also match with the optimized total ratios of SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>, N<sub>2</sub>O/SiO<sub>2</sub> and H<sub>2</sub>O/total solids in the mixture, which can be considered as alternative control parameters for the formulation process. The bulk density is more related to AS/FCC value. A geopolymer binder with a compressive strength of 24.7 MPa after curing for one day at 65°C and six days at room temperature was produced using this spent FCC waste.

## 1. Introduction

Spent fluid catalytic cracking (FCC) catalyst is a by-product from the petroleum industry used for improving the process efficiency [1]. The activity of this catalyst declines with time and when this happens, the “old inactive” catalyst is replaced by a “new active” catalyst [1]. This generates huge amounts of solid wastes; as an example, an oil refinery in Piura, northern Perú, discards about 1 400 ton of spent FCC every year as solid wastes. The total quantity of spent FCC generated worldwide is estimated to be about 170 000 t/year [1,2] and it will continue increasing as the environmental requirements of cleaner fuels increase [1]. Unfortunately, unless reuse or regeneration treatments are applied to reduce the quantity of spent catalyst discarded [1], after some cycles of regeneration, further regeneration may not be economically feasible, and the spent catalyst must be discarded in landfills [1]. The storage, transportation, treatment and final disposal of the spent catalyst require compliance with stringent environmental regulations because of their hazardous nature [1]. FCC contains mainly SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub> together with some minor impurities. The spent catalyst discarded additionally contains 7-



20% V+Ni, 15-25% coke, 7-15% Sulphur and 5-10% residual oil together with active metals (Mo and Co or Ni) [1].

The use of FCC as raw materials in the production of by-products is an attractive option from the environmental and economic points of view [1]. Besides of the preparation of abrasives, synthetic aggregates, glass-ceramics, and refractory bricks and cement [1], spent FCC has been used in the Portland clinker production [4]. Evaluation of this spent FCC as pozzolanic material in cement binder production [3,5] and cement mortars [6,7] have been carried out. Other researches have focused the fine aggregate replacement in concrete [8], and the improvement of its durability [9]. In transportation, FCC catalysts have been studied as filler and fine aggregates in asphalt mixtures [10,11]. More recently, preliminary evaluations of FCC catalysts for the development of geopolymers and alkali activated binders has been developed [2,12,13]. Geopolymers and alkali activated binders are binders developed as a friendly environmental alternative of Ordinary Portland Cement (OPC) [14] due to its reduced CO<sub>2</sub> emissions [15] and other properties [14,16]. Tashima et al. [12,13] synthesized stable geopolymer binders and mortars with compressive strengths in the range of 8–68 MPa using spent FCC catalyst. Alkali-activated mortars with up to 80 MPa of compressive strength were developed using this FCC waste [13]. In both studies, sodium silicate and sodium hydroxide were used to synthesize the FCC-based binders. Additionally, spent FCC has been evaluated to produce geopolymeric roof tiles [17] and concrete blocks [18].

An optimum concentration of the alkaline solution is required for an adequate activation of the solid precursor or raw materials [13,19,20], which is controlled by the silica modulus, sodium concentration and liquid alkaline solution/solid precursor weight ratio. In previous research, a strong dependence of compressive strengths values with the silica modulus has been observed [13] and other research suggest the existence of an optimum value [19,20,21]. It has been also determined that this FCC residue requires a solution of NaOH and waterglass for activation [12] and that there is an optimum Na<sub>2</sub>O concentration (in this case, expressed as a percentage in weight of the FCC in solid state) associated to an optimum silica modulus [13]. Other researchers suggest other parameters for the optimization of the formula for alkali-activated materials. For example, Varela et al. [22] proposed the used of total SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> and Na<sub>2</sub>O/SiO<sub>2</sub> molar ratios, which include the chemical compounds of both the alkaline solution and the solid precursor instead of the precursor alone. On the other hand, the concentration of the alkaline solution used for activation influences the bulk density of hardened geopolymer. Bumanis et al. [23] found that density of metakaolin-based geopolymers increase by increasing the activator's concentration because of the porous microstructure produced by evaporation of the free water in the material. The aim of this paper is to evaluate the influence of chemical composition parameters on the compressive strength and bulk density of the geopolymer binder using the spent FCC catalyst produced by a local oil-refinery plant in Piura, Perú. For this purpose, different range values of sodium concentration, silica modulus and the liquid/solid weight ratio were analyzed, using the compressive strength and bulk density as parameters of control. The use of this waste material would have two positive aspects for the environment, it could replace OPC in some construction applications and avoid disposing of the material in scarce landfills.

## 2. Experimental program

### 2.1. Materials

The spent FCC was supplied by Incablock SAC, a local concrete precast company. The chemical composition of the spent FCC catalyst was obtained by X-ray fluorescence spectroscopy (see results in table 1). The spent FCC catalyst has slightly more SiO<sub>2</sub> and less Al<sub>2</sub>O<sub>3</sub> than similar materials [e.g. 3,12,13], and its SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub> molar ratio is more than one. Lanthanum and vanadium oxides were identified as contaminants present in the zeolite structure during the catalyst synthesis [3]. The mineralogical composition was gathered with an X-ray diffraction (XRD) analysis. The total amorphous components represent between 64% to 66% of the total. Crystalline zeolite phases as Y-zeolite, and ZSM-5 zeolite are present in the catalyst residue. Less than 3% of other aluminosilicates such as mullite,

and quartz were also observed. The spent FCC has a specific gravity of  $2.6 \text{ g/cm}^3$ , measured using the standard water pycnometer method [24].

The spent FCC was subjected to a milling process to reduce the size of the particles and increase its reactivity [2,4,25-27]. The particle size range of the spent FCC was 1.3-150  $\mu\text{m}$  with a mean particle diameter of about 75  $\mu\text{m}$ . After milling, the particle size range was reduced to about 1.2-52  $\mu\text{m}$  with a mean particle diameter of about 22  $\mu\text{m}$ , which is slightly larger compared to what is reported in previous studies [3,12,13]. The water needed for a normal consistency of the FCC [28] was about 77% (or 0.77 mass ratio).

**Table 1.** Chemical composition of the spent FCC catalyst used in this study (wt%).

SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	MgO	CaO	Na <sub>2</sub> O	K <sub>2</sub> O	TiO <sub>2</sub>	La <sub>2</sub> O <sub>3</sub>	V <sub>2</sub> O <sub>5</sub>	P <sub>2</sub> O <sub>5</sub>	LOI	Others
49.61	46.23	0.70	0.19	0.09	0.70	0.06	0.70	2.93	0.91	0.20	1.01	2.52

The alkaline solutions (AS) were prepared by mixing sodium silicate (SS) and sodium hydroxide solution [12]. Sodium hydroxide pellets (99% purity) was dissolved in deionized water to prepare the sodium hydroxide solution and a commercial sodium silicate solution (water glass, 28% SiO<sub>2</sub>; 8% Na<sub>2</sub>O: 64% H<sub>2</sub>O) was used as provided. The AS were prepared at least 2 hours prior to their use to allow the solution to cool to room temperature ( $20 \pm 2^\circ\text{C}$ ). This step is necessary to avoid the premature setting of the geopolymer mixture and a reduction on the final properties [25,27].

### 2.2. Sample preparation and testing procedure

Geopolymer samples were prepared by mixing the dry spent FCC catalyst powder with the AS at approximately  $20^\circ\text{C}$ . The fresh paste was cast immediately in cubic molds (5 cm x 5 cm x 5 cm) and subjected to vibration for 1 minute to remove air voids, as recommended by ASTM C109 standard [29]. Samples were cured at  $65^\circ\text{C}$  for 1 day after which they were demolded and left at room temperature ( $20 \pm 1^\circ\text{C}$ ) for the rest of the time until testing. The compressive strength tests were performed by using a hydraulic testing machine MTS model Exceed 45.105 controlled by displacement, according to ASTM C109 standard [29]. The bulk density was estimated as a weight in air per volume unit; size of the samples was measured using a Vernier caliper. The bulk densities and compressive strengths of cubic samples were determined after 7 days. The reported values were the average of three samples.

### 2.3. Mixtures proportion

Three parameters were considered in the preparation of the alkaline activating solution: the silica modulus (Ms), NaOH concentration (NaOH) and liquid alkaline solution/solid precursor mass ratio (AS/FCC). Ms is related to the molar ratio Na<sub>2</sub>O/SiO<sub>2</sub> in the activating solution which has a significant effect on the strength of the geopolymers. This parameter determines the amount of soluble silicates present and it is crucial in controlling the dissolution rate and the gelation process during geopolymerization [30]. NaOH is the molar concentration of Na<sup>+</sup> of sodium hydroxide in the solution in mol/kg H<sub>2</sub>O and has relevance to assure the dissolution of the solid precursor. AS/FCC controls the consistency and helps in the workability of the fresh mixture and the uniformity of the final hardened product.

As shown in Table 2, the study was divided into three steps parts, described as follows:

(1) Analysis of the silica modulus (Ms). 15 M NaOH solution was combined with different quantities of the SS solution. Starting from an Ms value around 0.85, as suggested by [13], five mixtures were evaluated (SERIES A).

(2) Analysis of the NaOH concentration. For a fixed Ms molar ratio of 0.85, four samples with different NaOH molar concentrations of 7.5 M, 10 M, 12.5 M and 15 M were evaluated (SERIES B).

(3) Analysis of liquid alkaline solution/solid precursor ratio (AS/FCC). With both, the Ms and NaOH molar concentration fixed, the AS/FCC was varied from 0.8 to 2.0 to change the consistency of the mixture. Five mixtures for two series of 15M (SERIES C) and 10M (SERIES D) respectively were used to verify the influence of NaOH molarity on the AS/FCC value.

**Table 2.** Mix proportions of FCC geopolymeric samples used for evaluation.

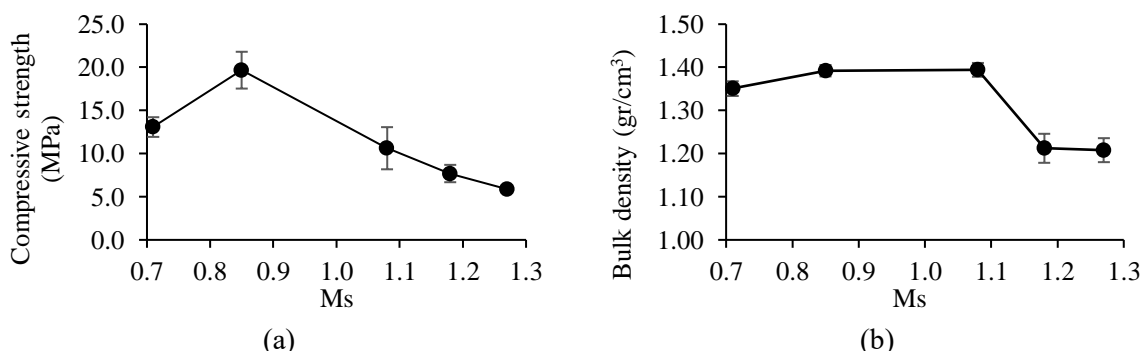
SERIE	NaOH solution (mol/L)	Ms (molar ratio)	AS/FCC (mass ratio)	SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub> (total) (molar ratio)	Na <sub>2</sub> O/SiO <sub>2</sub> (total) (molar ratio)	H <sub>2</sub> O/total solids (total) (wt.)
A1	15	0.71	1.10	2.39	0.33	0.49
A2	15	0.85	1.18	2.50	0.32	0.51
A3	15	1.08	1.32	2.68	0.30	0.55
A4	15	1.18	1.37	2.76	0.29	0.57
A5	15	1.27	1.42	2.84	0.28	0.59
B1	15	0.85	1.18	2.50	0.32	0.51
B2	12.5	0.85	1.26	2.50	0.32	0.57
B3	10	0.85	1.39	2.50	0.32	0.66
B4	7.5	0.85	1.58	2.50	0.32	0.79
C1	15	0.85	0.80	2.28	0.24	0.39
C2	15	0.85	1.00	2.39	0.28	0.45
C3	15	0.85	1.10	2.45	0.30	0.49
C4	15	0.85	1.18	2.49	0.32	0.52
C5	15	0.85	1.30	2.56	0.34	0.55
D1	10	0.85	1.10	2.36	0.27	0.56
D2	10	0.85	1.35	2.48	0.31	0.65
D3	10	0.85	1.39	2.50	0.32	0.66
D4	10	0.85	1.73	2.67	0.37	0.76
D5	10	0.85	2.08	2.84	0.42	0.86

Four additional parameters are included for the evaluation in table 2. Valera et al. [22] suggested that molar ratios of the mixtures need to include the SiO<sub>2</sub> and Na<sub>2</sub>O species provided by both the alkaline solution and the solid precursor. In this study, SiO<sub>2</sub>/Al<sub>2</sub>O<sub>3</sub>, Na<sub>2</sub>O/SiO<sub>2</sub>, Na<sub>2</sub>O/Al<sub>2</sub>O<sub>3</sub> and H<sub>2</sub>O/total solids ratios of all samples were calculated using the chemical concentration of the species in the final mixture. A possible correlation between the compressive strength and these parameters are evaluated.

### 3. Results and discussion

#### 3.1. Effect of silica modulus (Ms)

The results show that the compressive strength of the FCC based geopolymer binder increased with the Ms until 0.85. This optimum value can be identified in the figure 1a. Higher Ms values limits the dissolution of Si and Al species due to the lack of Na<sup>+</sup> [31]. For lower values of Ms, an excessively high concentration of NaOH resulted in premature precipitation of the geopolymer gel due to the excessive OH<sup>-</sup> ions [22,31]. The 0.85 Ms value is very close to what was obtained by [13], for a similar spent FCC residue.



**Figure 1.** Effect of the Ms modulus on (a) the compressive strength and (b) the density of FCC based geopolymer binder.

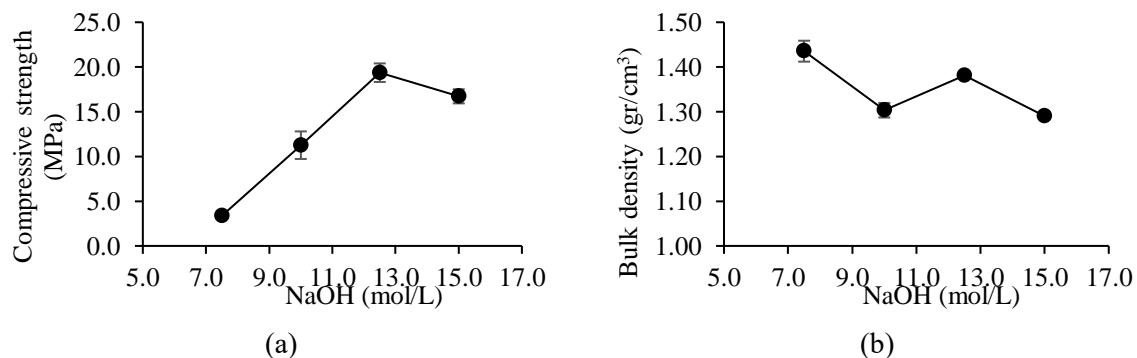
In respect to the bulk density, a decreasing tendency related to the optimum Ms can be observed (figure 1b), unless this variation is not clear. It can be attributed to the large amount of water (H<sub>2</sub>O/Na<sub>2</sub>O ratio) present for high Ms ratios. This may be explained since free water occupies a space in the volume

of the sample and as the water is not part of the crosslinked aluminosilicate network, it evaporates, leaving empty spaces which can be recognized as porosity with a low specific weight [30]. This behavior confirms that the bulk density is not affected by the Ms [32].

### 3.2. Effect of NaOH concentration

The compressive strengths of the FCC based geopolymer binders increased with increasing the NaOH molar concentration from 8 to 12.5 M in the mixture; when 15 M NaOH solution was used, the compressive strength remained relatively constant (only 5.3% increase) (figure 2a). A higher NaOH concentration may produce a higher compressive strength due to the better dissolution of the Si and Al species during the polymerization reaction, as found by other researches [30,31]. However, an excessive NaOH concentration can also dissolve the stable species formed initially, limiting the formation of the geopolymerization products and reducing the compressive strength [13].

As shown in figure 2b, the relationship between the bulk density and NaOH concentration is not clear. Again, water played a role only during the first stages of the geopolymerization until its elimination during the polymerization process, generating the porosity that explains the density.



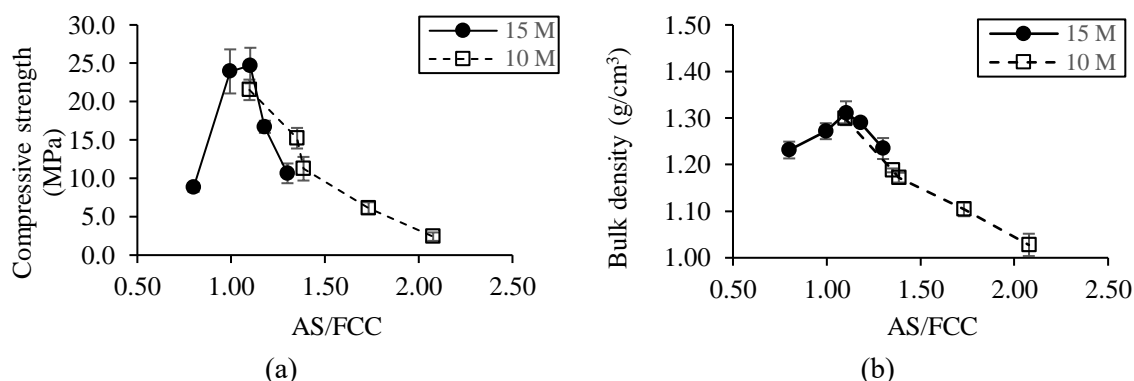
**Figure 2.** Effect of the [NaOH] on (a) the compressive strength and (b) the bulk density of the FCC based geopolymer binder.

### 3.3. Effect of liquid alkaline solution/solid precursor ratio (AS/FCC)

An optimum value of AS/FCC ratio of 1.10 produced the highest compressive strengths with both 10 M and 15 M NaOH concentration (figure 3a). For lower values of AS/FCC ratios, the mixtures were too dry and difficult to introduce in the molds. The low compressive strengths of the dry mixtures can be also attributed to the lack of enough NaOH or liquid medium in the mixture. For AS/FCC values higher than 1.20, the mixtures had a liquid consistency. However, a reduction of the compressive strength was observed for these mixtures which could be attributed to the excess of activator and the inhibition of the polymerization process [13].

In general, these results confirm that the workability of the mixture is important because it influences the compressive strength [18] and the AS/FCC value does not depend on the NaOH concentration. AS/FCC assures the adequate accommodation in molds and the homogeneity of the samples. This same behavior was observed in previous studies and confirms the attack of the precursor and the chemical combination of  $\text{Na}^+$ ,  $\text{OH}^-$  and silicate ions in the geopolymeric matrix [12]. In conclusion, an AS/FCC ratio between 1.0 to 1.37 produced mixtures with adequate consistency and workability that made them easy to accommodate in the molds without much effort.

The bulk density is more directly related to the AS/FCC ratio (figure 3b). For the 15 M NaOH alkaline solution, the optimum AS/FCC ratio produced the highest bulk density. Similar behavior can be observed for the 10 M NaOH solution, where the optimum AS/FCC ratio produced the lowest density. This confirms that AS/FCC value has an effect in the bulk density [33].



**Figure 3.** Effect of the AS/FCC weight ratio on (a) the compressive strength and (b) bulk density of the FCC based geopolymer binder.

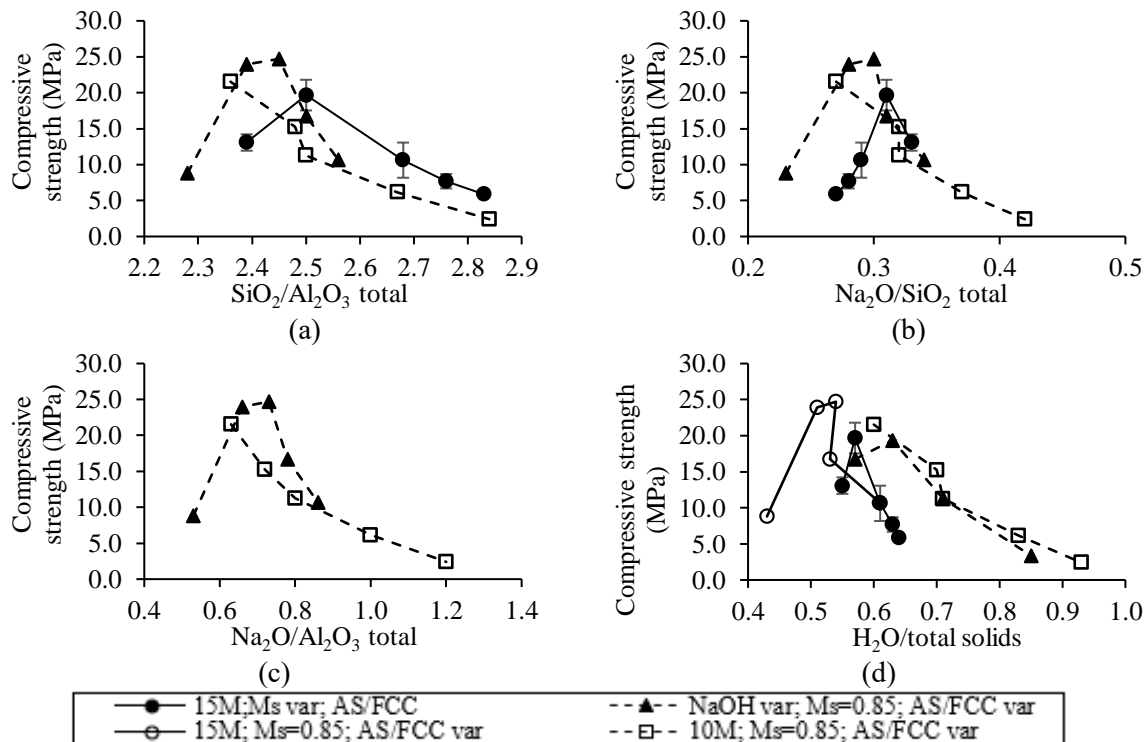
It can be concluded that the optimum formulation for geopolymerization of spent FCC catalyst requires an alkaline activator solution prepared with 15 M NaOH solution and a sodium silicate of Ms of 0.85, in a proportion of AS/FCC close to 1.1, approximately. The optimum AS/FCC has 17.25 % of  $\text{Na}_2\text{O}$  and 18.72% of  $\text{SiO}_2$ , that is close to the range suggested by [13]. The difference can be attributed to the type and quantity of the amorphous components and finesse of the precursor material, although complete data was not reported.

#### 3.4. Analysis of the total ratios $\text{SiO}_2/\text{Al}_2\text{O}_3$ , $\text{Na}_2\text{O}/\text{SiO}_2$ , $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ and $\text{H}_2\text{O}/\text{total solids}$

A very strong correlation between the compressive strength and the chemical composition on the final mixture can be observed in the figure 4. These parameters were calculated as the chemical species on the total mixture (alkaline solution and FCC residue). For the three key factors evaluated previously (NaOH concentration, Ms and AS/FCC) there is a correspondence to the optimum values of the total ratios. A  $\text{SiO}_2/\text{Al}_2\text{O}_3$  final molar ratio between 2.4 and 2.5 produces the highest 7 days compressive strength (figure 4a) which corresponds to both the optimum Ms and AS/FCC values. In the same way, an optimum  $\text{Na}_2\text{O}/\text{SiO}_2$  final molar ratio close to 0.30 (figure 4b) corresponds to both the optimum Ms and AS/FCC values. These results are consistent with previous researches. For example, [6] found optimum  $\text{SiO}_2/\text{Al}_2\text{O}_3$  and  $\text{Na}_2\text{O}/\text{SiO}_2$  total molar ratios for fly ash and rice husk bark ash based geopolymers that produced the maximum compressive strengths. For the  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  molar ratio the optimum values are in a range between 0.6 and 0.7. Additionally,  $\text{H}_2\text{O}/\text{total solids}$  ratios between 0.4 and 0.7 were related to the highest compressive strength (figure 4d), which is consistent with the range of 0.4 and 0.6 values for  $\text{H}_2\text{O}/\text{FCC}$  obtained by [12] for a similar FCC. Similar finding was reported by [34] in a class C fly ash geopolymer mortar, reaching a compressive strength of 10.29 MPa at a  $\text{H}_2\text{O}/\text{total solids}$  ratio of 0.40.

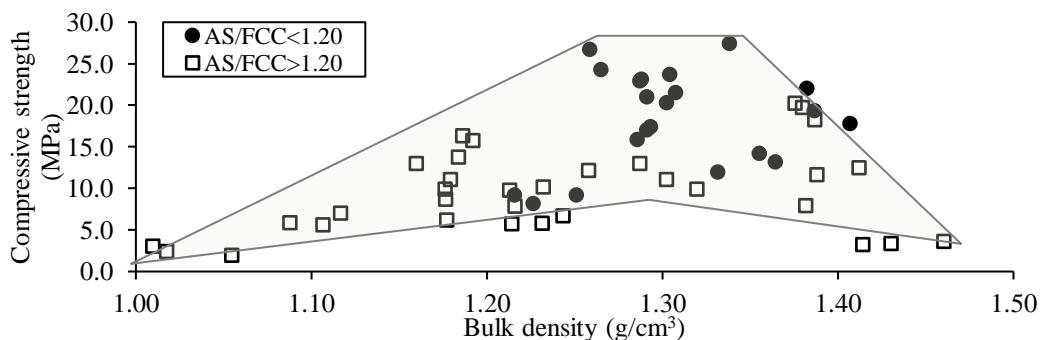
#### 3.5. Compressive strength and bulk density

The bulk densities and compressive strengths of the mixtures at the age of 7 days are plotted in figure 5. Bulk densities of geopolymer binders are between 1.08 and 1.47  $\text{g}/\text{cm}^3$  which are relatively low compared to other geopolymer binders [35,23]. There is a region where the maximum compressive strength is related to a range of density between 1.25  $\text{g}/\text{cm}^3$  and 1.35  $\text{g}/\text{cm}^3$ . For lower values, the compressive strength is lower too. This region is related to high values of AS/FCC that represents an excess of solution for a certain weight of FCC residue. This excess of solution is also associated to a high amount of water that produces a porous microstructure, reducing both the density and the compressive strength. But, when this high AS/FCC value is associated to high NaOH concentrations, the bulk density increases due the presence of the total solid species in the mixture, but the excess of  $\text{Na}^+$  cations inhibits the formation of the stable species during the polymerization process reducing the compressive strength. These values correspond to the region of densities higher than 1.35  $\text{g}/\text{cm}^3$ .



**Figure 4.** Influence of the total ratios in the final mixture (including both the FCC residue and the alkaline solution) on the compressive strength (a)  $\text{SiO}_2/\text{Al}_2\text{O}_3$ , (b)  $\text{Na}_2\text{O}/\text{SiO}_2$ , (c)  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  and (d)  $\text{H}_2\text{O}/\text{total solids}$ .

For low AS/FCC values, there is low amount of water in the mixture that contributes to increase the density but also limits the compressive strength because of the reducing in the workability of the mixture. This lack of workability does not allow the polymerization process. In this region, the key factors evaluated in previous sections play an important role for the development of the compressive strength. This finding confirms that, considering an adequate range of AS/FCC value, the compressive strength is not related to the density but to the quality of bonds formed in the dimensional network of the geopolymer [33] as a consequence of the Ms and NaOH concentration [35,23].



**Figure 5.** Comparison of the bulk density and the compressive strength in FCC based geopolymer binders for all the mixtures evaluated in this research.

#### 4. Conclusions

The compressive strength depends on some key parameters of formulation such as Ms, NaOH concentration and AS/FCC value. This study allowed reaching a maximum 7 day-compressive strength

of 24.7 MPa when a Ms modulus of 0.85, 15 M NaOH solution and a AS/FCC value of 1.10 was used. The results also indicate that total ratios in the mixture ( $\text{SiO}_2/\text{Al}_2\text{O}_3$ ,  $\text{Na}_2\text{O}/\text{SiO}_2$ ,  $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$  and  $\text{H}_2\text{O}/\text{total solids}$ ) that includes the species of both the solid precursor and the alkaline solution, can be used to control the optimum formulation for geopolymers production. The bulk density of the geopolymer binders were between 1.08 and 1.47  $\text{g}/\text{cm}^3$ , which is lower than other geopolymer binders. The bulk density has a strong correlation to the AS/FCC value used in the formulation. Finally, it can be concluded that spent FCC catalyst from the oil refinery industry is suitable to produce geopolymer binders. This new application for the residue may contribute to reduce the environmental impact of its disposal.

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