Compression and characterization of alkali-activated loess

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Abstract. The concept of sustainable development continues to move forward and natural resources, wastes or by-product materials can be used as full or partial replacement in building constructions due to their environmental benefits. This research investigates the effective use of easily available and low cost natural loess in the preparation of geopolymer green materials. Na2SiO3 and NaOH were used as the reactive solution to dissolve silica and alumina in the mixture composed of loess and fly ash particles, and the compressive strength performance and the microstructure characterization on the synthesized geopolymer pastes were carried out. Experimental results demonstrated that loess is a suitable geopolymer precursor with good compressive resistance and high degree of binding and compact microstructure.

1. Introduction

During the past decade, geopolymer have become an extensively researched binding material because of its technological, environmental and economic advantages as compared to Portland cement. Indeed, the use of Ordinary Portland cement in the construction industry is under critical review due to the high amount of carbon dioxide that is released into the atmosphere during cement production [1]. Joseph Davidovits, French materials scientist invented the term “Geopolymer” in 1979, attributed this term to the amorphous to semi-crystalline tri-dimensional alumino-silicates that can be formed at low temperature and short time by alkali reaction with naturally occurring alumino-silicates solid materials [2]. The mechanism of this activation involves three major reactions (a) Dissolution of aluminate and silicate monomers when OH− in the alkaline activator breaks the bond of alumino-silicates bonded with oxygen in the source material (b) Polycondensation of silica and alumina monomers to form dimers, which in turn react with another monomer to build polymer (c) Crystallization occurs by the precipitation of reaction products [3]

Various source materials rich in Silicon (Si) and Aluminum (Al) were developed and activated by alkaline liquids to produce a binder alternative to Portland with satisfactory results [4-9]. Loess is defined as an eolian yellow light-coloured fine grained accumulation which is mainly composed of silt mineral and clay particles. It is characterized by an open structure and is very often found around the world, including central Asia, central Europe, northwestern and central USA, South America, northern Russia, and interior Alaska, and has been studied as natural material since many years by renowned authors [10, 11]. According to the research by Stevens et al. [12], the most complete and thickest loess deposits are found in China, in the provinces of Shanxi, Shaanxi, and Gansu; and the dust deposition and the formation of continuous loess in China's loess plateau began 22 years ago [13].
As stated by the current studies, there is little information in the literature on loess systems. A systematic study was conducted to investigate the compressive strength behavior of full and partial addition of loess into fly ash, followed by the microstructure characterization of the synthesized geopolymer paste.

2. Materials and method

2.1. Materials
Natural loess was supplied from Shaanxi Province in China and the low calcium classified as class F fly ash according to ASTM C618-08a specification [14] were used as raw materials. Sodium silicate Na$_2$SiO$_3$ solution and sodium hydroxide NaOH in pellets form were used as alkali activators in the preparation of the geopolymer.

2.2. Mix design
The mixture proportions (Table 1) adopted in this study is composed of 170g of NaOH, 1060g of Na$_2$SiO$_3$, 60g of plasticizer and 700g of water as a total volume of the tested specimens. The sodium silicate (Na$_2$SiO$_3$) solution was mixed with sodium hydroxide (NaOH) in pellets of 98% purity to form 14M molarity concentration. The proportions of loess were taking by a consecutive addition ratio into fly ash of 10% in the range of 10-100%.

| Specimen Label | Loess (%) | Fly ash (%) | Loess (g) | Fly ash (g) | Alkaline Solution (g) | Water (g) | Plasticizer (g) |
|----------------|-----------|-------------|-----------|-------------|-----------------------|-----------|-----------------|
| Lr-10          | 10        | 90          | 420       | 3780        | 170                   | 1060      | 700             |
| Lr-20          | 20        | 80          | 840       | 3360        | 170                   | 1060      | 700             |
| Lr-30          | 30        | 70          | 1260      | 2940        | 170                   | 1060      | 700             |
| Lr-40          | 40        | 60          | 1680      | 2520        | 170                   | 1060      | 700             |
| Lr-50          | 50        | 50          | 2100      | 2100        | 170                   | 1060      | 700             |
| Lr-60          | 60        | 40          | 2520      | 1680        | 170                   | 1060      | 700             |
| Lr-70          | 70        | 30          | 2940      | 1260        | 170                   | 1060      | 700             |
| Lr-80          | 80        | 20          | 3360      | 840         | 170                   | 1060      | 700             |
| Lr-90          | 90        | 10          | 3780      | 420         | 170                   | 1060      | 700             |
| Lr-100         | 100       | 0           | 4200      | 0           | 170                   | 1060      | 700             |

2.3. Precursor structural characterization

The chemical composition analysis of Loess and FA by x-ray diffraction (XRD) was performed and the results of these materials are presented in Table 2. The results showed that loess covers a large range of particle sizes mainly composed of silica and alumina, with 60.33% of Silicon (SiO$_2$) and 12.57% of Aluminum (Al$_2$O$_3$). Similarly, the FA used in this study is mainly composed of 60.70% of Silicon (SiO$_2$), 24.72% of Aluminum (Al$_2$O$_3$) and 6.90% of ferric oxide (Fe$_2$O$_3$).

To investigate the composition and microstructure of the materials, 90% of the crushed specimen and the two raw materials were taken for further investigations. Microstructural characterization was performed with the aid of a Hitachi S4700 scanning electron microscope.

| Composition | SiO$_2$ | Al$_2$O$_3$ | Fe$_2$O$_3$ | TiO$_2$ | MnO | CaO | MgO | K$_2$O | Na$_2$O | P$_2$O$_5$ | SO$_3$ | LOI |
|-------------|---------|-------------|------------|---------|-----|-----|-----|-------|--------|---------|-------|-----|
| Loess Content (%) | 67.33 | 12.57 | 2.72 | 0.63 | 0.05 | 7.57 | 2.48 | 2.40 | 2.10 | 0.15 | — | — |
| FA Content (%) | 60.70 | 24.72 | 6.90 | — | — | 0.70 | 1.13 | — | — | 1.50 | 2.35 | — | — |

Table 2. Loess and FA chemical composition
2.4. Mixing and curing procedure
NaOH was mixed together with the Na$_2$SiO$_3$ solution based on predefined proportions as the reagents to dissolve the silica and alumina in the source material particles. Afterwards, loess and fly ash were first dried mixed, and afterwards mixed with the alkaline activator, polycarboxylate superplasticizer and additional water using a rotating pan mixer. CASTING OF MOLDS WITH SIZE 70.7 mm CUBE WAS USED AND THE SPECIMENS WERE PLACED ON A VIBRATION TABLE TO REMOVE ENTRAPPED AIR.

Two replicate samples i.e. 6 cubes for each mixture ratio were cured for 7 and 14 days respectively. After 24 hours rest period, the specimens were placed at environment chamber under 60°C, 50% Relative Humidity (RH). This temperature value was chosen because [15, 16] found that the curing temperature, the time and the sequential addition of the synthesized parameters have a big impact on the strength. Fig 1 shows the homogeneous loess paste and the specimens under curing conditions.

![Figure 1. Homogeneous loess paste and specimens under curing conditions](image)

3. Results and discussion

3.1. Compressive strength properties
The curves related to the compressive load-displacement relationship of loess-based geopolymer paste specimens at different curing time (Fig. 2) were analyzed and the compressive strengths values were computed. The standard deviation value varied from ±2 and the curve shape remain linearly elastic throughout the test until failure. The average compressive strengths of all the tested specimens are in the range of 14.54 MPa-9.26 MPa. The average compressive strength of the full loess-based geopolymer specimen at 7 days curing period is 12.29 MPa and 10.98 MPa at 14 days curing time.
3.2. Effect of loess/fly ash ratio variation on the compressive strengths

It is evident that Lr/FA ratio greatly influences the compressive strengths of the geopolymer paste (Fig. 3). It can be seen from Lr/FA=90% that the higher the value of the ratio, the higher the value of the strength but this comparison is countered by the ratio Lr/FA=20% which presents approximately the same compressive strength with reduction up to 0.22 MPa at 7 days curing time. Moreover, the compressive strength at 14 days curing time also shows a contrast, with the strength increasing up to 1.62 MPa for the specimen with 20% ratio but in both cases, the ratio of Lr/FA=50% presented the lowest strength values with 11.82 MPa and 9.26 MPa at 7 and 14 days respectively.

Apparently one can adopt Lr/FA=90% value as a limit if considering solely strength as the basis of the design, and the presence of loess in the geopolymer paste is notable since it is comparably cheaper than fly ash and a good filler material, it also provides maximum economic solution. From this result, following the research works of [17-18] on the strength of geopolymer paste and mortar specimens, one can notice that the compressive strengths results of the geopolymer paste in this study is largely acceptable.
3.3. Microstructure characterization

Fig. 4 shows the micromorphology of the natural loess, fly ash, and loess-based geopolymer for 90% loess ratio. By comparing the SEM images of the natural loess and fly ash, we noticed that they present different particles size. From the figure, it was observed that the loess-based geopolymer presents more or less good structure. This indicates how the compact microstructure was rendered in the mixture with the increasing ratio of 90% loess and 10% fly ash. This demonstrates the high degree of binding and also the compact microstructure of the synthesized paste, which agrees well with the properties of geopolymer source materials.
Figure 4. SEM micrographs of the natural loess, fly ash, and loess-based geopolymer
4. Conclusion

In this study, high volume of available local loess with partial replacement of fly ash was used to synthesize green geopolymer pastes. Based on the experimental results the following conclusions were drawn:

a) Loess consists essentially of silicon and aluminum.

b) The average compressive strengths of up to 12.29 MPa and 10.98 MPa, can be obtained from the full loess-based geopolymer specimens respectively at 7 and 14 days curing time.

c) The ratio of 20% and 90% replacement represent the most significant ratio for the mix design preparation and the compressive strengths of up to 14.32 MPa and 14.54 MPa, respectively can be obtained within 7 days curing period.

d) The ratio of 10%, 30% and 80% can be used in the mixture when expecting an average compressive strength value of 13.44 MPa. Similarly, the ratio of 40%, 60% and 70% like the ratio of 100% loess can be used in the mix design for an average compressive strength value of 12.38 MPa at 7 days curing period.

e) The addition of loess and fly ash of up to 50% reduces suitably the compressive strength.

f) The microstructure characterization demonstrated the high degree of binding and compaction of the developed geopolymer pastes.

From these results, one can concluded that, loess can be used in terms of geopolymer source materials with acceptable mechanical performance, and can also be applied in the energy sector as a greener alternative to Portland cement.

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