

Waste-to-Value Strategies in Material Science: A Case Study of Geopolymers from Fly Ash and Eggshells



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1.1 Abstract:

Geopolymers are the new advanced materials with high thermal insulation used in construction field. These materials have gain much attention in modern world due to the sustainability and green nature to help environment from more damage. In this research paper, the successful formation of geopolymer material is reported by using available fly ash and egg shell powder. The slurries with flyash, egg shell powder with sodium silicate were prepared. The maximum compressive strength of this material was marked at 11.3925Mpa, bulk density of that sample was 1.67 gcm⁻³, for a geopolymer having 10% fly ash and 60 % egg shell powder.

1.2 Introduction:

Talking about the natural effects of Portland cement, it has given a great push to the sideeffects of its expanded use. Cement in this era is considered the most widely used material in the world. On average, approximately 1 ton of cement is produced annually (Van Den Heede & De Belie, 2012). On yearly production, almost 2.6 billion tons of cement is produced noted in 2006 (Van Oss, 2006). And its manufacturing was increased by 54% from 2000 to 2006 (Tam et al., 2009), where the CO₂ emission is increased by 42% (560Mt) reaching 1.88Gt₂ in 2006 (IEA) (Tam et al., 2009). Its manufacturing is supposed to be increased at the rate of 0.8-1.2% annually because of its expanding use, increasing consumption, and population growth (Hoagland et al., 2011). Its production process involves quarrying raw material, transporting and crushing, clinker calcination at 1400~1450°C, grinding requires 3.2GJ energy/ton. Moreover, cement production increases 5~7% of globally (Habert et al., 2015; Van Den Heede & De Belie, 2012).

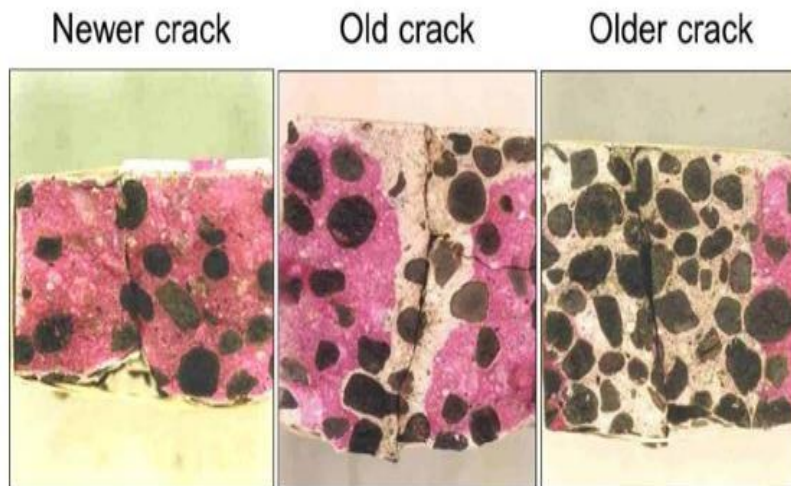
For CO₂ reduction, World Business Council for Sustainable Development and IEA has given a road map in 2009. It says to outline a possible way to halve the global CO₂ by 2050. It states that the cement industry should reduce its emission by 18% and this reduction does not imply a linear reduction by the same percentage. It is an ambitious vision, yet changes must be practical, and realistic. Some low-carbon cements are developing like Calera, Calix's cement, and Geopolymer, which are still in the development phase (Tam et al., 2009).

The major advancement of time in the beneficial use of industrial was is the activation of aluminosilicate materials such as fly-ash, blast-furnace slag, and metakaolin solutions. They reduces, the unfriendly

effects of cement. Alkali-activated binders have many names in the literature: alkali-activated cement (Rožek et al., 2021), geopolymer, inorganic-polymer, hydroceramic, or low-temperature aluminosilicate glass. They are also called polysialates because of the silicon-oxygen-aluminium polymeric structure (Rožek et al., 2021). Geopolymers are defined as a tri-dimensional network of -Si-O-Si(Al)- bonds, synthesized by mutating solid sources of SiO₂ and Al₂O₃ with an alkaline solution. Geopolymers are synthesized by using aluminosilicate precursor such as clay, metakaolin (Jingkun Yuan, 2016), natural soil (Syed Farrukh Alam Zaidi E. U.-u.-R., 2017), bottom ash (J. Temuujin, 2010) (HuiXu, 2010) (Musaad Zaheer NazirKhan, 2016), rice husk ash (Gediminas Kastiukas, 2016) (M.F.Zawrah, 2016), and volcanic ashes. And as the pollution increase is gaining attention these decades all over the world as different organizations are working on it. For that purpose, new high properties constructional material and need of reducing waste for future are considered a "green" ideas and in that condition using ashes by activating them will be helpful. Other than high chemical, physical and mechanical properties these materials are also low-carbon emitting, economical and long-life materials as compared to cement. Cement has a carbonation effect due to which its lifetime reduced and lasts for about 50-100 years. Concrete structures are exposed to aggressive environments so their durability is one of the major concerns (J. Chi, 2002). Carbonation is the reaction of hydration products dissolve in water in pore and CO₂ in the air reduces pH of concrete. pH reduction from 12.6 to 9 increases cracks development and decreases concrete durability (V. Papadakis, 1992). "Carbonation testing provides a

means with which the inspector can determine the extent of carbon dioxide infiltration into the concrete. The common revealer used is phenolphthalein."

"When phenolphthalein comes into contact with high pH (>10) concrete the solution shows as bright pink. When the solution comes into contact with low pH



(<10) the solution shown no color change and the

Figure 1: Image of Cracked Concrete Samples After Carbonation Testing. Note the Older Crack and the Amount of Carbonation in the Concrete

concrete can be considered carbonated. Cracking in concrete allows the open air to penetrate further into the concrete surface. Cracking near reinforcing steel allows the pH in the concrete surrounding the reinforcing to be reduced that much sooner (manual, 2011)."

Fly ash is divided into two categories based on origin and composition. **Class F** is made by burning bituminous or anthracite coal having a composition $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \geq 70\%$. Whereas **class C** is obtained by burning sub-bituminous or lignite coal having a composition $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 \geq 50\%$ (Burduhos Nergis et al., 2018). Fly-ash has many hazardous elements like boron, cadmium, nickel, and lead that causes asthma, respiratory ailments, and eye-diseases.

Where for its disposal ash-ponds are made that further contaminate underground water.

With this great idea of waste utilization eggshells is another widely produced waste. Eggs are largely used in bakery-products salad-dressings and fast-foods that produce tons of waste a high management cost daily. Almost 250,000 tons of waste is produced annually. But its amazing composition of CaCO_3 and low-porosity can be a blessing if used properly. Chemically (by-weight) it has 94% calcium-carbonate, 1% magnesium-carbonate, 1% calcium-phosphate and 4% organic matter (Faridi & Arabhosseini, 2018).

1.3 Methods and procedures:

Geopolymer mortar was prepared by using fly ash produced at Kot Addu Power station, eggshell powder from waste and sodium silicate (Na_2SiO_3). The egg shell waste used in covers more than 11% of the waste of Pakistan (Ashfaq Ahmed Jhatial, 2019). The density of Na_2SiO_3 was 1.37g/cm^3 and it contained 61% of water, 9% of sodium oxide and 30% SiO_2 . The particle size of eggshell powder was reduced by ball milling down to 200 mesh size $\sim 74\mu\text{m}$. The reason for reducing its size was to get higher surface area so that would increase its reactivity. Therefore, more reactivity is possible with finer particle size (Ehsan UlHaq, 2016).

1.4 Preparation of samples:

In this work four compositions were prepared by varying the amount of fly ash and eggshell powder. Three samples of each composition were prepared. **Table 1** shows the prepared compositions and sample designations. ESG stands for Experimental Sample Group and followed by number. The remaining composition of samples depends on the amount of sodium silicate, sodium hydroxide and water contents.

All the ingredients were mixed properly and proper quantity of slurry was prepared into metal molds ($L=2''$, $W=2''$, $H=2''$). Subsequently, the foamed samples were then demolded and then allowed to further dry in open air for three days. Basic steps are illustrated in **fig. 2**.

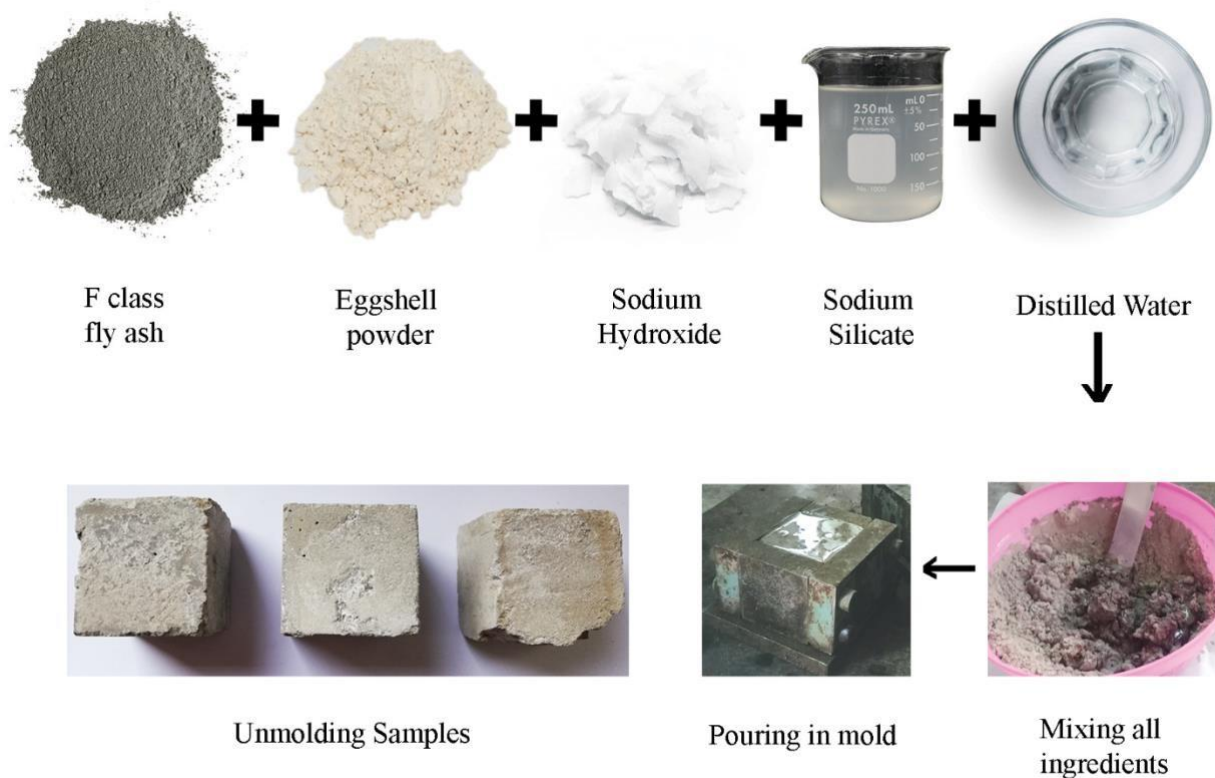


Figure 2: Basic steps in the preparation of natural soil based geopolymer mortar samples

1.5 Characterization methods:

Bulk density of all samples were calculated according to BS EN 12390- 7:2009. True density of mortar samples was calculated by ASTM D854-14 protocols. And then porosity percentage was evaluated by using the equation given below as **Eq. 1**:

$$\text{Percentage of Porosity} = \{1 - (\text{Bulk density} / \text{True density}) \} \times 100 \quad (1)$$

Compressive strength of experimental samples was tested on Universal Testing Machine (UTM) with that of 9.8kN according to the ASTM C109 protocols then average of the reading were calculated.

Table 1 Compositions of the geopolymer foam samples.:

Sample ID	Fly ash wt. %	Eggshell powder wt. %	Sodium hydroxide wt. %	Sodium silicate wt. %
ESG1	70	0	7	18
ESG2	50	20	7	18
ESG3	30	40	7	18
ESG4	10	60	7	18

1.6 Results and discussions:

1.6.1 Bulk density and porosity:

Bulk density of resultant mortars are plotted in **fig.3**:

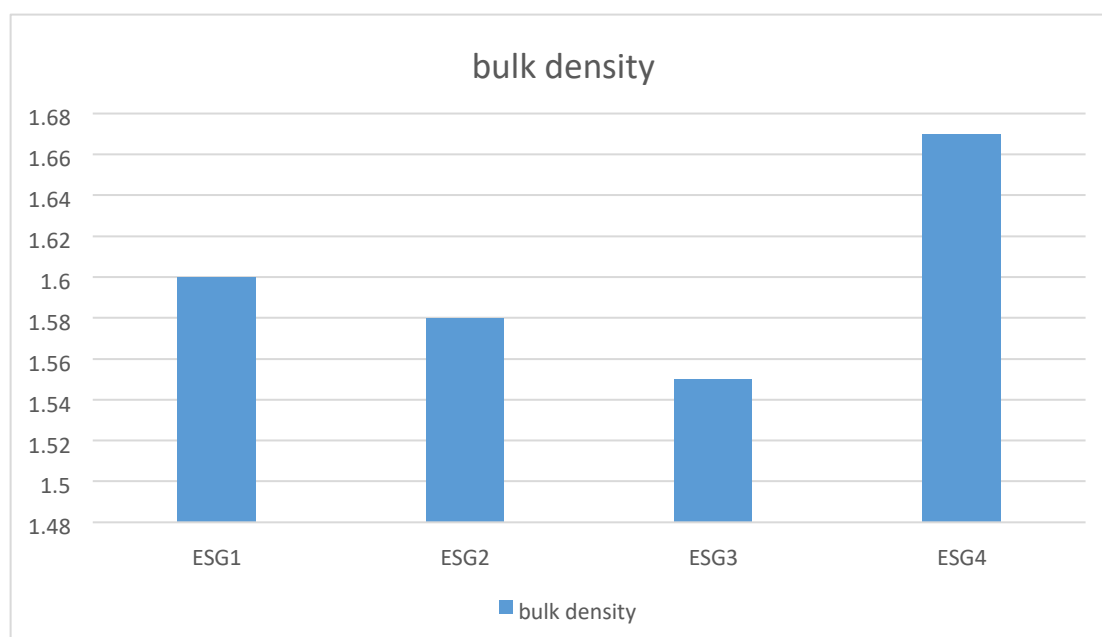


Figure3: Bulk density of ESGs.

Whereas, the %total porosity of these samples are shown in fig.4:

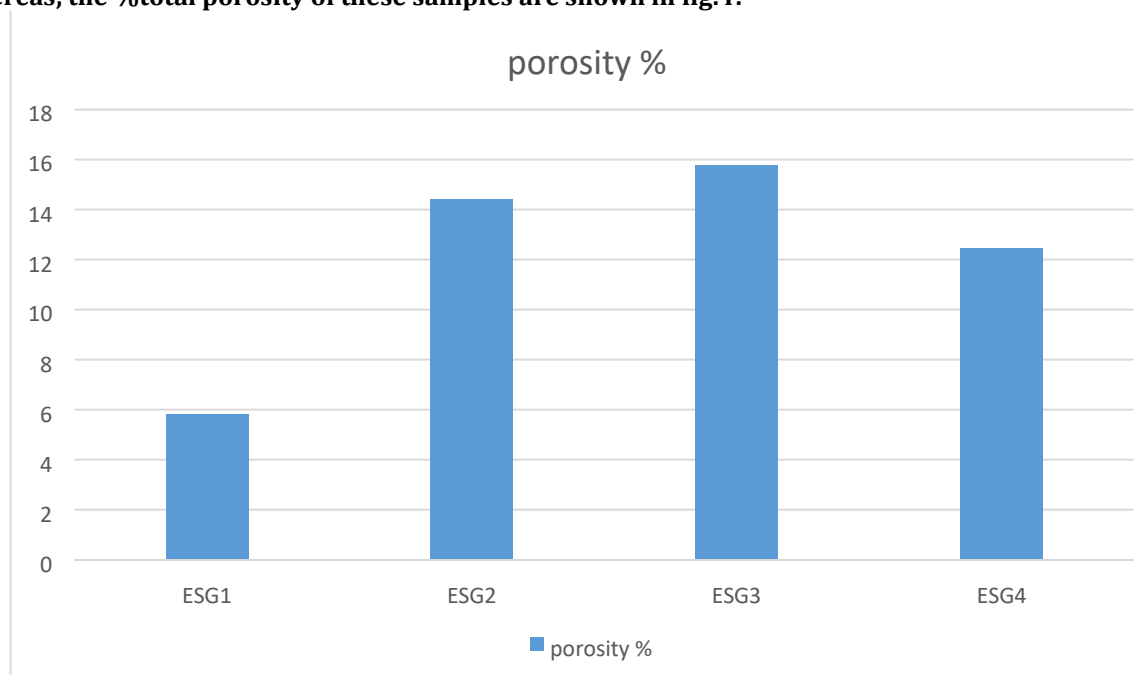


Figure3: Porosity percentages of ESGs.

As a general result, Bulk density and porosity percentages are in inverse relationship (Sanosh Kunjalukkal Padmanabhan, 2015) (V.Vaou, 2010). As the value of bulk density goes up the percentage of porosity moves in opposite direction.

The value of bulk density falls up to ESG3 and rises at ESG4 for where in correspondence to this porosity percentage gradually rises till ESG3 due to reduced geopolymerization (Syed Farrukh Alam Zaidi E. U.-u.-R., 2017) and falls at ESG4.

1.7 Physical and mechanical properties:

Mechanical properties in compression mode, amount of fly ash and amount of porosity are interrelated in **Fig. 5:**

The compressive strength decreases from ESG1 to ESG2 due to increase in porosity but then gradually increases due to reduction in porosity percentage. There is an increasing trend in porosity percentage from ESG1 to ESG3 and then it reduces at ESG4 due to increase in geopolymerization. Maximum

compressive strength of 12.44MPa achieved at ESG1 because of maximum degree of geopolymerization and followed by ESG4 with that of 11.3925MPa. The achieved compressive strength value is greater than many researches done before on Geopolymers (Alina Ioana Badanoiu, 2015) (Chengying Bai, 2016) (Syed Farrukh Alam Zaidi E. U.-u.-R., 2017). The compression with previous researches are given in **table.2:**

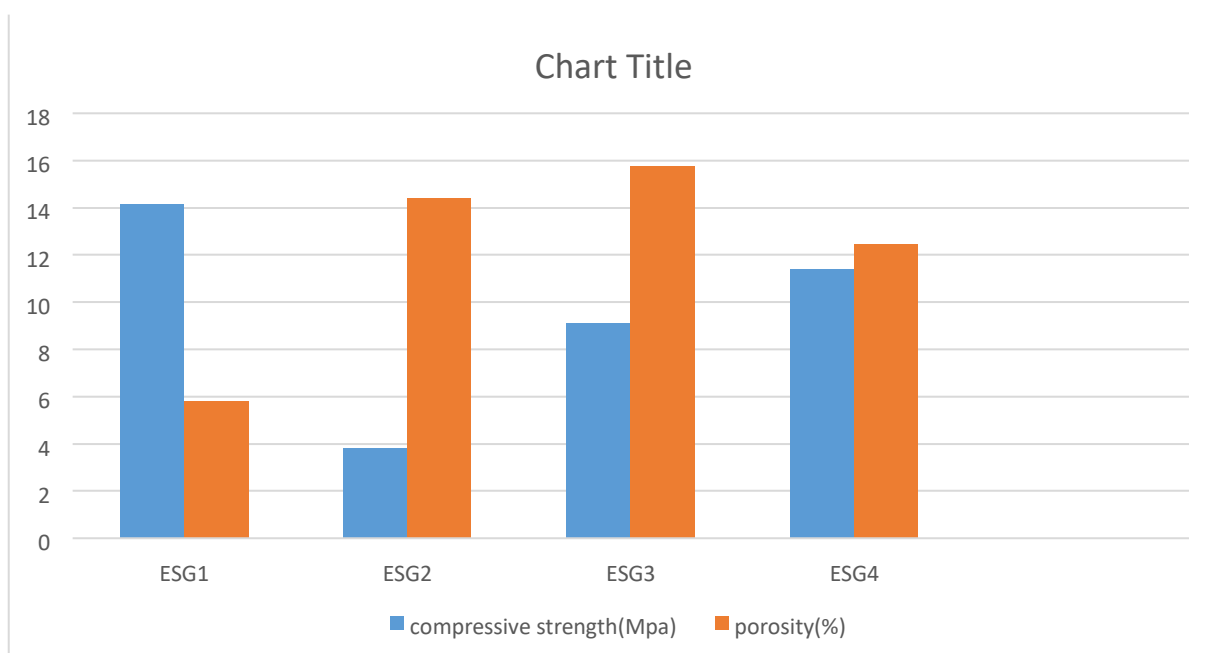


Figure 5: Interrelation of mechanical strength, porosity.

Minimum compressive strength is in ESG2 due to low degree of polymerization. Compressive strength of all samples are given in **Table.3.**

Table 2: Comparison of maximum compressive strength with some others work

Maximum compressive strength (MPa)	References
2.10	(Alina Ioana Badanoiu, 2015)
3.1	(Chengying Bai, 2016)
2.41	(Syed Farrukh Alam Zaidi E. U.-u.-R., 2017)
12.344	Current work

In low porous samples, maximum amount of geopolymerization gives maximum compressive strength. Drastic change in the compressive strength from ESG1 to ESG2 is because of the changing composition of flyash and eggshell powder.

Table 3: Physical and mechanical properties of samples.

Sample ID	Bulk density(g/cm ³)	Apparent density(g/cm ³)	True density(g/cm ³)	Compressive strength (MPa)	Porosity (%)
ESG1	1.6	1.78	1.79	12.44	5.8
ESG2	1.58	1.67	1.8	3.797	14.4
ESG3	1.55	1.6	1.84	9.114	15.76
ESG4	1.67	1.8	1.9	11.39	12.42

1.8 Conclusion:

In this experiment, the samples were successfully made from the abundantly available two wastes i.e. fly ash and egg shell powder. The samples were allowed to be cured in open air after demolding the geopolymer. The degree of geopolymerization was excellent in ESG1 but it reduced in ESG2 and the consistently increased. Sample ESG1 is proved to be the optimum sample with maximum properties, sample contained no egg shell powder. But the very next best results are derived from ESG4 having 60% of egg shell powder.

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