Research Article

Study on the Production Factors in the Process of Production and Properties of Fly Ash-Based Coarse Aggregates

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An optimization study was carried out for the sustainable production of coarse aggregates from fly ash and alkaline solution, considering the combined effect of alkaline solution and production process. The trial mixes during the process of producing the artificial aggregates were designed through Taguchi's experimental design method. The combined effect of alkaline solution (geopolymerisation) and production process (pelletization factors) along with engineering properties of the produced coarse aggregates was evaluated using response indices at different curing ages. Furthermore, the influence of each individual factor of geopolymerisation and pelletization on the engineering properties was determined through grey relational analysis to identify the most influencing factors in the production of coarse aggregates. The results obtained from grey relational analysis indicate that the properties of produced aggregates are governed mostly by geopolymerisation. It is also observed that water content of 20% by mass of fly ash is found to be essential for the suitable production of coarse aggregates and factors such as Na_2O content and curing regime improved the engineering properties.

1. Introduction

The statistic depicts global concrete production is approximately 5.3 billion cubic meters per year [1]. Typically, ordinary concrete contains about 70–80% aggregate, 12% cement, and 8% mixing water by mass [2]. On the other hand, the use of aggregates in the construction of aggregates is enormous. In the year 2019, global demand for construction aggregates was more than 51.7 billion tonnes, which is estimated to grow 5.2% annually. In India alone, it is predicted that consumption of aggregates is more than 5 billion metric tonnes in the year 2020 [3]. At present, the consumption rate of natural aggregates in global level has reached an alarming level. Hence, there is a necessity of producing an alternative and sustainable material which could replace the natural aggregates in concrete.

Many researchers have reported on the aggregates production using fly ash with suitable binding materials through artificial process [4]. In the production process, it is noted that different techniques are available such as agglomeration, expansion (bloating), pressing and crushing, etc., [4–6]. However, it is observed that the pelletization process is well developed agglomeration technique for producing artificial aggregates [4, 7–9]. However, the adoption of pelletization process in producing the artificial aggregates is still not being adopted widely by the construction industry and hence the production cost of artificial aggregates is found to be high as compared to the cost of natural aggregates.

For artificial fly ash aggregates it has been reported that production and properties of the aggregates mainly depend on the raw materials, size of the particles in the raw materials, type of binder, specific surface area, the wettability of particles, and production process involved [4, 9–14]. However, researchers reported that the engineering properties of produced fly ash aggregates are mainly influenced by the type and composition of binder used in the production process along with the dosage of the binder [12–14].

From the literature it is to be noted that the production process can be controlled in a better way with a disc type pelletizer as compared to other types of pelletizers such as drum or cone pelletizers [4, 11]. Furthermore, it is reported by the researchers that, in the pelletization process, different factors such as water content during pelletization, the angle of pelletizing disc, speed of pelletizing disc, and duration of the pelletization are the most influencing factors that affect the production process of aggregates [4, 11, 12]. The produced aggregates are subjected to different hardening methods such as sintering, normal water curing, cold bonding, steam curing, and autoclaving [4, 8, 10, 12–14].

The alkali activator solution, which acts as an activator in the geopolymerisation process, plays an important role in the surface hydrolysis of the raw material particles [15]. The alkaline solution with different types [15–19], concentrations [16–24], raw material to solution ratio [17, 25–27], and curing regime [15, 23, 24, 27, 28] has a significant influence in the geopolymerisation process. However, there are a limited number of studies reported in the literature on the utilization of alkaline solutions for the production of artificial fly ash aggregates, whereas the factors responsible for fly ash aggregates production through pelletization process, combined with the factors of geopolymerisation, are very much essential to understand the efficient and effective production.

To investigate the combined effect of geopolymerisation and pelletization factors on the properties of artificial fly ash aggregates, an experimental study is very much essential which could be carried out with the help of design of experiments. Various optimization techniques are available such as factorial designs [11, 29–33], central composites [34–36], response surface methodology [11, 35, 36], and Taguchi method [11, 37, 38]. Taguchi's experimental design method is one of the best optimization methods defined by Genichi Taguchi which allows experimental trial runs to a small number and also to identify the influence of factors selected [11, 29]. Considering all the factors mentioned above, this experimental investigation was carried out to determine the combined effect of geopolymerisation and pelletization factors in the sustainable production of artificial aggregates using Taguchi's experimental design method followed by grey relational analysis.

1.1. Significance of the Study.

The natural aggregates consumption is increasing rapidly because of the faster rate of infrastructure development worldwide. However, the mining of natural aggregates is creating a serious environmental imbalance. In the present situation, the challenge before the world is to have innovative and less carbon footprint materials for the construction industry. Hence, there is a need for an alternate material that can substitute the natural aggregate. Using artificial methods to produce substitute materials from industrial waste or byproducts will solve the disposal problem and at the same time, it is going to save natural resources. The use of artificially produced fly ash aggregate will gain significant importance for infrastructure development which can replace the naturally available materials.

The production of the artificial aggregates is having different methods as per requirements or the end use in the products, whereas many researchers reported on the process with different raw materials as well as the different binders for the production of artificial aggregates. The present study is concentrated on the production process by pelletization technique and alkaline solution content and on characteristics of produced coarse aggregate simultaneously.

2. Materials and Methodology

2.1. Materials.

Locally available fly ash was used in the production of artificial fly ash aggregate. The fly ash had a specific gravity of 2.20 and a specific surface area of $260.30 \, m^2/kg$. The chemical compositions are presented in Table 1 and the particle size distribution of fly ash analysed in the laboratory is presented in Figure 1. As per IS 3812 (Part 1) 2013 classification [39], fly ash has been classified as class F type. The laboratory grade sodium silicate solution (Na_2SiO_3) with alkali ratio (SiO_2/Na_2O) of 3.3 (8.0% Na_2O, 26.5% SiO_2, 65.5% H_2O by mass) and sodium hydroxide (NaOH) flakes of 98% purity were used to prepare the alkaline solution.

2.2. Experimental Programme.

The production of artificial aggregates is significantly affected by several factors such as type of alkali content and its dosage, raw material and its composition, and also the process of pelletization. In the present investigation, the raw material is fly ash and alkaline solution. To study the relative influence of several factors in the production process and aggregates properties, the following experimental programme was carried out.

The most influencing factors related to alkali content in geopolymerisation process and the factors related to geopolymerisation were identified as Na_2O content, SiO_2/Na_2O ratio, water content, and curing methods [22–24, 26]. An increase of Na_2O content and SiO_2/Na_2O ratio in alkaline solution will lead to higher level of binding property in the geopolymer [22–26]. The initial trial mixes were carried out with different composition of Na_2O content from 3% to 7% (percentage of the mass of fly ash), water content from 18% to 22% (percentage of the mass of fly ash), and SiO_2/Na_2O ratio from 0.3 to 0.5. This alkaline solution was then sprayed to the pelletization disc. It was observed that, with increase in Na_2O content (from 3 to 7%) and SiO_2/Na_2O ratio (from 0.3 to 0.5) with constant water content (21%), the alkaline solution was found to be highly viscous. This can be attributed to high content of solids in the solution [19, 40]. Hence, in the present study alkaline solution composition is restricted to 4 to 6% of Na_2O content along with the varying SiO_2/Na_2O ratio from 0.3 to 0.5 and water content from 19% to 21%.

Furthermore, factors related to pelletization process such as the angle of pelletizing disc, speed of pelletizing disc, water content, and duration of the pelletization were identified [4, 7, 11]. The angle and speed of pelletizing disc were varied from 35° to 55° with every 10° interval and 30 to 50 RPM with every 10 RPM interval, respectively. It is also observed from

TABLE 1: Chemical compositions of fly ash.

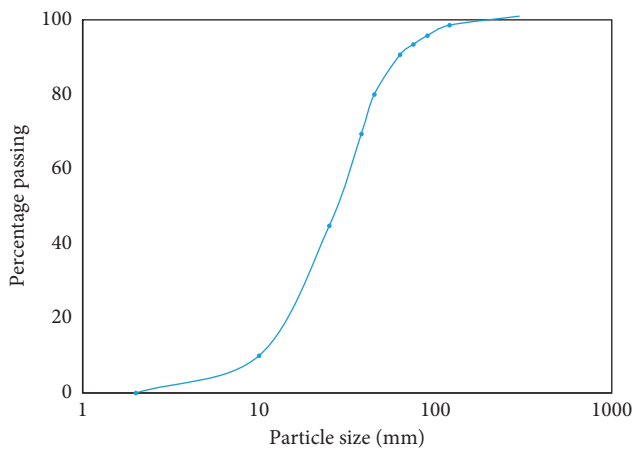| Chemical constituents	(%)
SiO_2	60.65
Al_2O_3	28.62
Fe_2O_3	3.95
MgO	1.84
CaO	1.70
SO_3	1.26
Na_2O	1.11
K_2O	0.11
LOI	2.6

FIGURE 1: Particle size distribution of fly ash.

the literature, for the formation of pellets, a minimum of 10 to 15 minutes is needed [7] and 12 to 18 minutes were selected with an interval variation of 3 minutes in the present study.

Although water content is a factor where both geopolymerisation and pelletization are getting affected, in the present study it was restricted; 19 to 21% of water content (% is with respect to mass of fly ash) was selected based on the previous study [41]. The water added in the preparation of sodium hydroxide solution and water present in the sodium silicate solution together reference as the water content in the alkaline solution.

After the production of aggregates, it is subjected to different curing conditions as follows: (i) curing at ambient temperature ($28 \pm 2°C$), (ii) heat curing at $80°C$ for 24 hours [15, 23, 24, 27, 42], and (iii) solution curing for 30 minutes [10] which will improve the properties of aggregates.

The objective of the present study is concerned with the investigation on the effect of geopolymerisation factors and pelletization factors as a combined factor in the aggregates production using fly ash as a raw material with alkali solution. To optimize the mixtures of fly ash and alkali solution in the production of artificial aggregates and to evaluate the interaction with the study parameters in the production and engineering properties of produced aggregates, the following design of experiments was used.

2.3. Design of Experiments with Taguchi's Orthogonal Array. Experimental design was carried out using Taguchi's orthogonal array. This orthogonal array was identified based on the factors and the levels which influence the most as described in the previous section and the same is presented in Table 2. In this phase the pelletization factors are allied with geopolymerisation factors and were investigated as the combined effect. The same was carried out with L27 (3^7) Taguchi orthogonal array to study the engineering properties of the artificial aggregates. The essential variables for each experimental trial run are labelled from TMC 1 to TMC 27 and they are presented in Table 3.

In the second phase, the response index for individual factor was analysed with the help of Minitab software to evaluate the response indices for all the selected factors from the experimental test results. Further, grey relational analysis of a statistical approach was analysed to understand the effect of combined factors from their responses all together in the production of sustainable aggregates.

2.4. Alkaline Solution Preparation. The required quantity of sodium hydroxide (NaOH) flakes was dissolved in normal tap water to prepare NaOH solution. The alkaline solution was prepared by mixing both NaOH solution and Na_2SiO_3 solution such that required proportions of Na_2O content (%), water content (%), and SiO_2/Na_2O ratio are maintained as per each trial mix provided in Table 3. With required proportion as specified in Table 3, alkaline solution is prepared and preserved in a closed container and allowed to cool, before using it in the production process.

2.5. Sustainable Aggregates Production. A laboratory scale disc pelletizer was used in this study [41, 42]. Pelletization was carried out in the following steps:

(i) The required proportion of alkaline solution was prepared

(ii) The necessary adjustment was made to the pelletizer

(iii) Weighed fly ash was transferred to the pelletizing disc

(iv) Required alkali solution was sprayed to the disc pelletizer directly within first three minutes during the pelletization process

2.6. Curing of Aggregates. In the present investigation artificially produced fly ash (APFA) aggregates were subjected to different curing conditions as per experimental programme and the details of each curing condition are as follows:

Ambient curing (A): the APFA aggregates through the pelletization process are kept at a temperature of $28 \pm 2°C$ and relative humidity of 80% and were characterised at 14, 28, and 56 days.

Heat curing (H): the APFA aggregates through the pelletization process are allowed to set for 24 hours in ambient temperature conditions. After that, fly ash aggregates are subjected to a temperature of $80°C$ for 24

TABLE 2: Different individual factors and their values considered in the study.

Factors	L1	L2	L3
Na_2O content (%)	4	5	6
SiO_2/Na_2O ratio	0.3	0.4	0.5
Water content (%)	19	20	21
Speed of pelletizing disc (RPM)	30	40	50
Angle of pelletizing disc (°)	35	45	55
Duration of pelletization (minutes)	12	15	18
Curing regime	A	H	S

L: level; A: ambient temperature; H: heat cured; S: solution cured.

TABLE 3: Combined factors and values used in the production trial runs.

Trial runs	Factors						
	Na_2O content	SiO_2/Na_2O ratio	Water content	Speed of pelletizing disc	Angle of pelletizing disc	Duration of pelletization	Curing regime
TMC 1	4	0.3	19	30	35	12	A
TMC 2	4	0.3	19	30	45	15	H
TMC 3	4	0.3	19	30	55	18	S
TMC 4	4	0.4	20	40	35	12	A
TMC 5	4	0.4	20	40	45	15	H
TMC 6	4	0.4	20	40	55	18	S
TMC 7	4	0.5	21	50	35	12	A
TMC 8	4	0.5	21	50	45	15	H
TMC 9	4	0.5	21	50	55	18	S
TMC 10	5	0.3	20	50	35	15	S
TMC 11	5	0.3	20	50	45	18	A
TMC 12	5	0.3	20	50	55	12	H
TMC 13	5	0.4	21	30	35	15	S
TMC 14	5	0.4	21	30	45	18	A
TMC 15	5	0.4	21	30	55	12	H
TMC 16	5	0.5	19	40	35	15	S
TMC 17	5	0.5	19	40	45	18	A
TMC 18	5	0.5	19	40	55	12	H
TMC 19	6	0.3	21	40	35	18	H
TMC 20	6	0.3	21	40	45	12	S
TMC 21	6	0.3	21	40	55	15	A
TMC 22	6	0.4	19	50	35	18	H
TMC 23	6	0.4	19	50	45	12	S
TMC 24	6	0.4	19	50	55	15	A
TMC 25	6	0.5	20	30	35	18	H
TMC 26	6	0.5	20	30	45	12	S
TMC 27	6	0.5	20	30	55	15	A

hours. Further, these aggregates are kept in ambient temperature conditions and were characterised at 14, 28, and 56 days.

Solution curing (S): the APFA aggregates through the pelletization process are first allowed to set for 24 hours in ambient temperature conditions. After that, fly ash aggregates are dipped in sodium silicate solution for 30 minutes. Further, these fly ash aggregates are kept in ambient temperature conditions and were characterised at 14, 28, and 56 days.

2.7. Testing on Aggregates. The pelletization efficiency in the aggregate production is calculated using equation (1). The particle size distribution in the production was determined using standard set of sieves (IS 383–2016) [43]:

$$\text{efficiency of pelletization} = \frac{\text{weight retained on the IS sieve no. 480}}{\text{total weight of materials used}} * 100. \tag{1}$$

The APFA aggregates were assessed for basic engineering properties such as water absorption, aggregate impact value, and aggregate crushing value as per IS 2386 [44, 45]. Crushing strength of individual pellet was estimated using a compression testing machine (ring type unconfined compression test apparatus) and crushing strength (σ) of fly ash aggregate was calculated from the following equation [7]:

$$\sigma = \frac{2.8 * P}{\pi * x^2}, \tag{2}$$

where P represents the failure load on the pellet and x is the size of pellets.

3. Experimental Results

Sustainable aggregates are artificially produced as explained in aggregate production section with trial runs as described in experimental program and they were evaluated for their production efficiency, particle size distribution, and engineering properties.

The experimental results were obtained at end of 14, 28, and 56 days; however, for the sake of brevity only 56 days' results are discussed in the subsequent sections.

3.1. Efficiency and the Particle Size Distribution of the Aggregate.
The pelletization efficiency of the produced aggregates with respect to water content was estimated using equation (1) and it is presented in Figure 2. It can be observed from Figure 2 that the efficiency is increased from 77.96% to 96.41% as water content increased from 19 to 21%.

The distribution of aggregates size in the production of fly ash aggregates with respect to different water content used is compared with nominal size of 20 mm graded aggregates as per IS 383–2016 [43] and presented in Figure 3. From Figure 3, it is observed that production of aggregates has a significant influence on the particle size distribution in production process. However, the APFA aggregates particle size distribution is within the limits of IS 383–2016 standards [43].

3.2. Specific Gravity and Bulk Density.
The APFA aggregates which are subjected to different curing conditions were assessed for specific gravity, bulk density, and percentage of voids. The average value of apparent specific gravity, saturated surface dry specific gravity, loose and compacted bulk density of aggregates, and percentage of voids for different fly ash aggregates subjected to curing conditions are presented in Table 4.

3.3. Aggregates Impact Value.
The response indices are determined from the experimental test results and the response index relationship for aggregate impact value with combined factors is presented in Figure 4. From Figure 4, it can be observed that, with increase in Na_2O content from 4% to 6%, aggregate impact value is improved from 27.88% to 22.78% at 56 days. Similarly increase in SiO_2/Na_2O ratio from 0.3 to 0.4 has improved the aggregate impact value from 26.71% to

25.47%, further increase of SiO_2/Na_2O ratio from 0.4 to 0.5 has marginal effect, and heat curing of artificially produced aggregates has significantly improved the aggregate impact value 23.69% when compared to aggregate impact values of 28.74% and 25.58% for ambient cured and solution cured aggregates at 56 days. The aggregate impact value gets marginally improved from 25.83% to 24.98% at 56 days with an increase in water content from 19% to 20%; further increase in the water content from 20% to 21% was found to have an adversarial effect on the aggregates impact value that decreased from 24.98% to 27.21% at 56 days. It is also noted from Figure 4 that the aggregates impact value has marginally improved from 25.93% to 25.59% due to the increase in the speed of pelletizing disc from 30 RPM to 50 RPM. Further increase in the angle of pelletizing disc from 35° to 55° and duration of pelletization of 12 to 18 minutes has shown the negligible improve on the aggregate impact values from 26.04% to 25.96% and 26.08% to 26.25% at 56 days, respectively.

3.4. Aggregate Crushing Value.
The experimental test results are used to determine the response indices and the response index relationship for aggregate crushing value with combined factors is presented in Figure 5. From Figure 5, it can be observed that, with the geopolymerisation factors such as Na_2O content increase from 4% to 6%, aggregate crushing value is improved from 27.21% to 23.92% at 56 days. Similarly, increase in SiO_2/Na_2O ratio from 0.3 to 0.5 has marginally improved the aggregate crushing value from 26.72% to 25.33% at 56 days. It is also observed that heat curing and solution curing of aggregates have significantly improved the aggregate crushing value of 23.37% and 25.34%, respectively, at 56 days when compared to the aggregates crushing value 28.72% at 56 days of ambient cured fly ash aggregates. Aggregates crushing value has declined from 25.13% to 26.35% at 56 days due to increase in the water content in the trial mixes. However, from Figure 5, response indices of pelletization factors indicate that the duration of pelletization of 12 to 18 minutes has improved the aggregates crushing value from 26.62% to 24.17% at 56 days. Further, increase in speed of pelletizing disc from 30 RPM to 50 RPM and angle of pelletizing disc from 35° to 55° has negligible improvement on the aggregate crushing value from 26.01% to 25.93% and 25.93% to 25.98%, respectively, at 56 days.

3.5. Crushing Strength of Individual Pellets.
The response indices are calculated from the experimental test results and the response index relationship for the crushing strength of individual pellets with combined factors is presented in Figure 6. From Figure 6, it can be observed that, with the geopolymerisation factors such as the Na_2O content increase from 4% to 6%, crushing strength of individual pellets is improved from 3.04 MPa to 3.79 MPa at 56 days. Similarly, increase of SiO_2/Na_2O ratio from 0.3 to 0.5 has improved the crushing strength of individual pellets from 3.24 MPa to 3.57 MPa at 56 days and heat curing has significantly improved the pellets crushing strength value to 3.81 MPa when
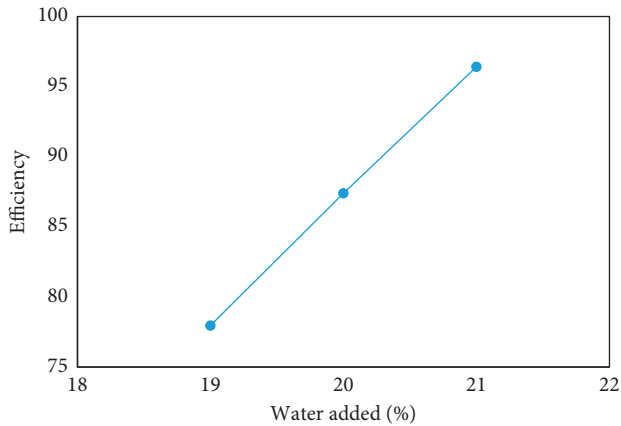
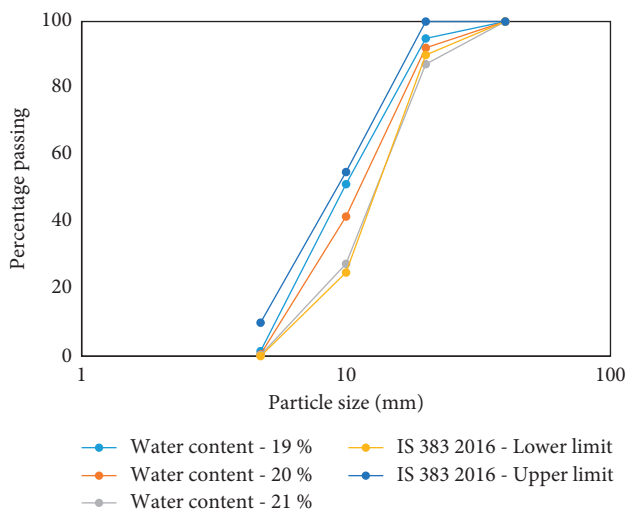FIGURE 2: Pelletization efficiency with respect to water content.

FIGURE 3: Particle size distribution of produced aggregates along with IS 383–2016 limits.

TABLE 4: Average specific gravity and bulk density of artificial fly ash aggregates subjected to different curing conditions.

Curing regime	Specific gravity		Bulk density (kg/m³)	
	Apparent	Saturated surface dry	Loose	Compacted
Ambient	1.95	1.65	1100	1210
Heat	1.97	1.62	1050	1170
Solution	1.97	1.71	1110	1220

compared to crushing strength of individual pellets values 3.08 MPa and 3.28 MPa of ambient cured and solution cured aggregates at 56 days. Aggregate crushing strength of individual pellets has declined from 3.50 MPa to 3.21 MPa at 56 days with increase in the water content from 19% to 21%. It can be also understood from Figure 6 that pelletization factors response indices indicate a trivial improvement in pellets crushing strength from 3.36 MPa to 3.49 MPa due to the increase in the duration of pelletization from 12 to 18 minutes. However, increase in the angle of pelletizing disc from 35° to 55° and speed of pelletizing disc from 30 RPM to

50 RPM has shown the negligible improvement on the pellets crushing strength from 3.38 MPa to 3.39 MPa and 3.39 MPa to 3.41 MPa, respectively, at 56 days.

3.6. Water Absorption. The experimental test results are used to determine the response indices and the response index relationship for water absorption with combined factors is presented in Figure 7. From Figure 7, it is found that, with geopolymerisation factors such as Na_2O content from 4% to 6%, water absorption value improved from 11.37% to 9.04% and with increase in SiO_2/Na_2O ratio from 0.3 to 0.5 the water absorption also reduced from 10.91% to 9.92% at 56 days. It is also observed that solution curing of artificially produced aggregates has significantly reduced the water absorption value to 8.42% when compared to water absorption values of 10.58% and 12.44% for ambient cured and heat cured aggregates at 56 days. However, increase in speed of pelletizing disc from 30 RPM to 50 RPM and change in the angle of pelletizing disc from 35° to 55° have improved the water absorption of aggregates marginally from 10.47% to 9.82% and 10.76% to 10.26%, respectively, at 56 days. It is observed that duration of pelletization of 12 to 18 minutes has the water absorption values of 10.42% to 10.68% which has negligible effect on water absorption of aggregates.

4. Grey Relational Analysis

Sahoo et al. have presented the advantages of grey relational analysis in categorizing the most influencing parameters with respect to the material properties [46]. In this investigation, experimental test results on the APFA aggregates were considered as responses and grey relational analysis was carried out as follows.

4.1. Determination of Grey Relational Generations. The aggregate properties results such as aggregate impact value, aggregate crushing value, crushing strength of individual pellets, and water absorption are considered as responses for the grey relational analysis. In this analysis, standardization of responses is carried out such that the lower the obtained value for the produced aggregates the better for aggregate impact value, aggregate crushing value, and water absorption, whereas the higher value obtained is better for crushing strength of individual pellets of APFA aggregates. Grey relational generation was obtained with the experimental values to scale of 0–1 and it was calculated using equation (3). Computed grey relational generations on the responses are presented in Tables 5–7 at different curing ages.

$$x_i = \frac{\max y_i(k) - y_i(k)}{\max y_i(k) - \min y_i(k)}, \tag{3}$$

where $x_i(k)$ = value after the GRG; $\min y_i(k)$ = smallest value of $y_i(k)$ for the kth response; and $\max y_i(k)$ = largest value of $y_i(k)$ for the kth response.

4.2. Assessment of Grey Relational Coefficients and Grey Relational Grades. The assessment of Grey relational
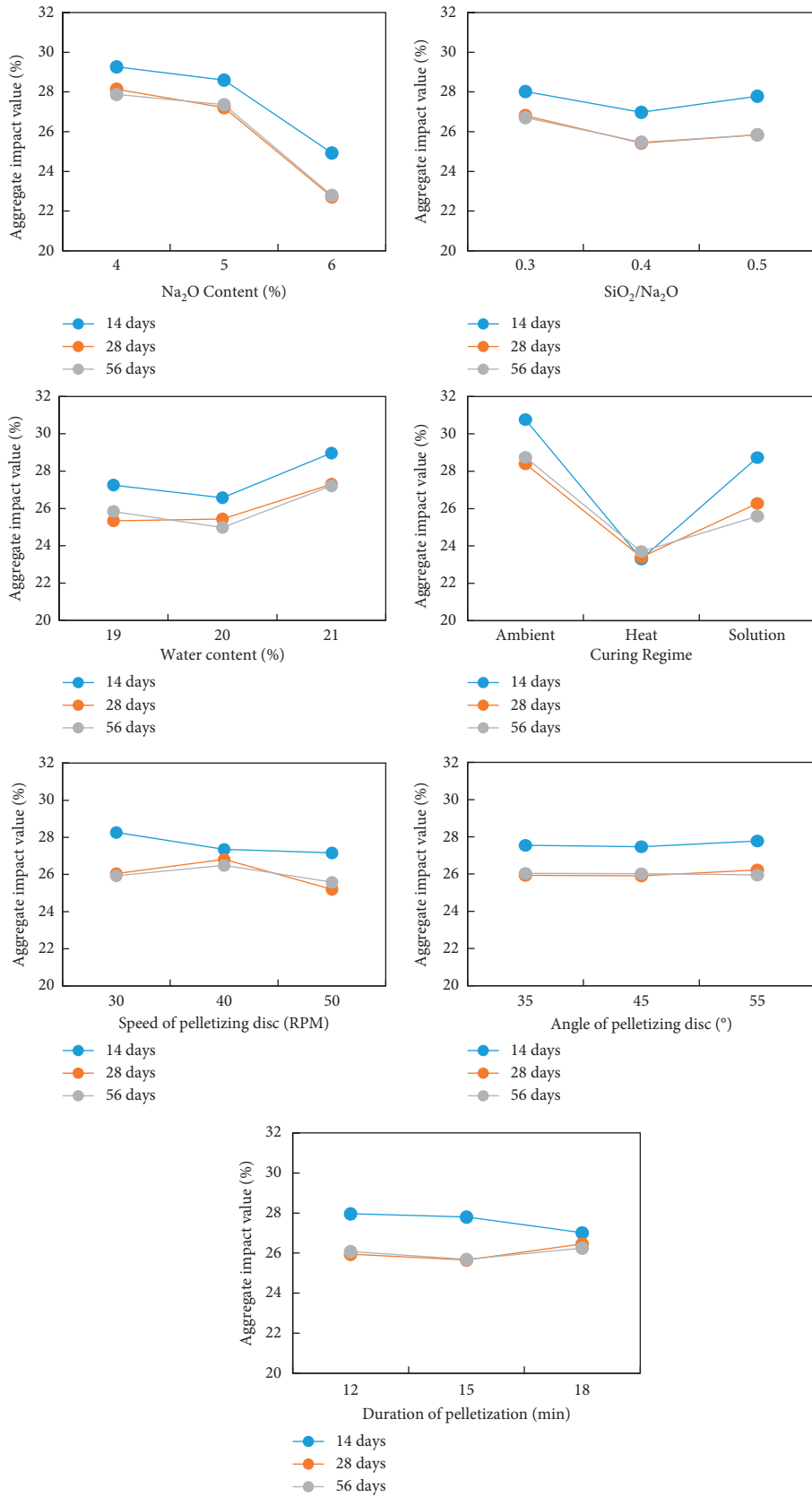
FIGURE 4: Response index relationship for aggregate impact value with combined factors.
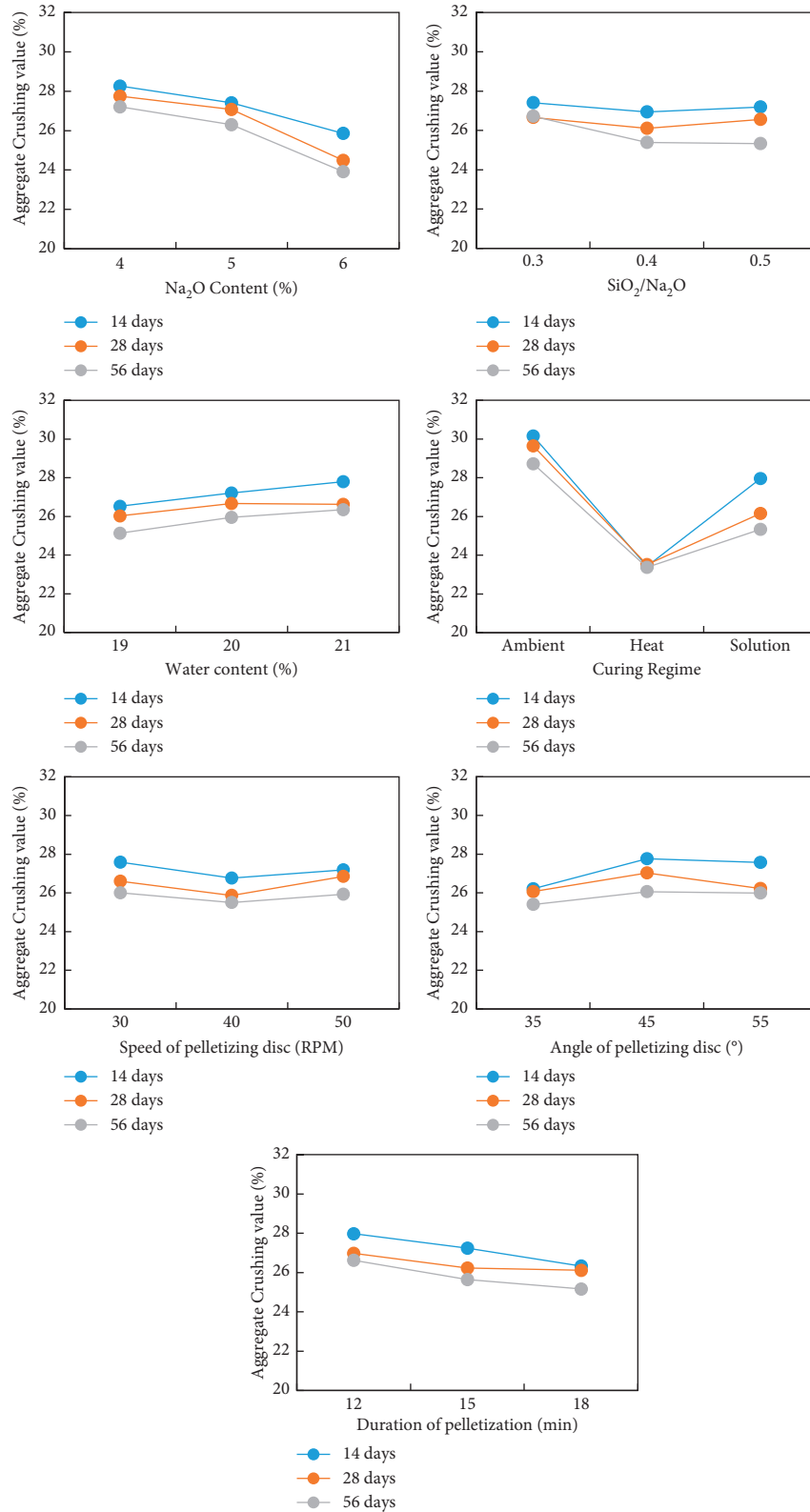
FIGURE 5: Response index relationship for aggregate crushing value with combined factors.
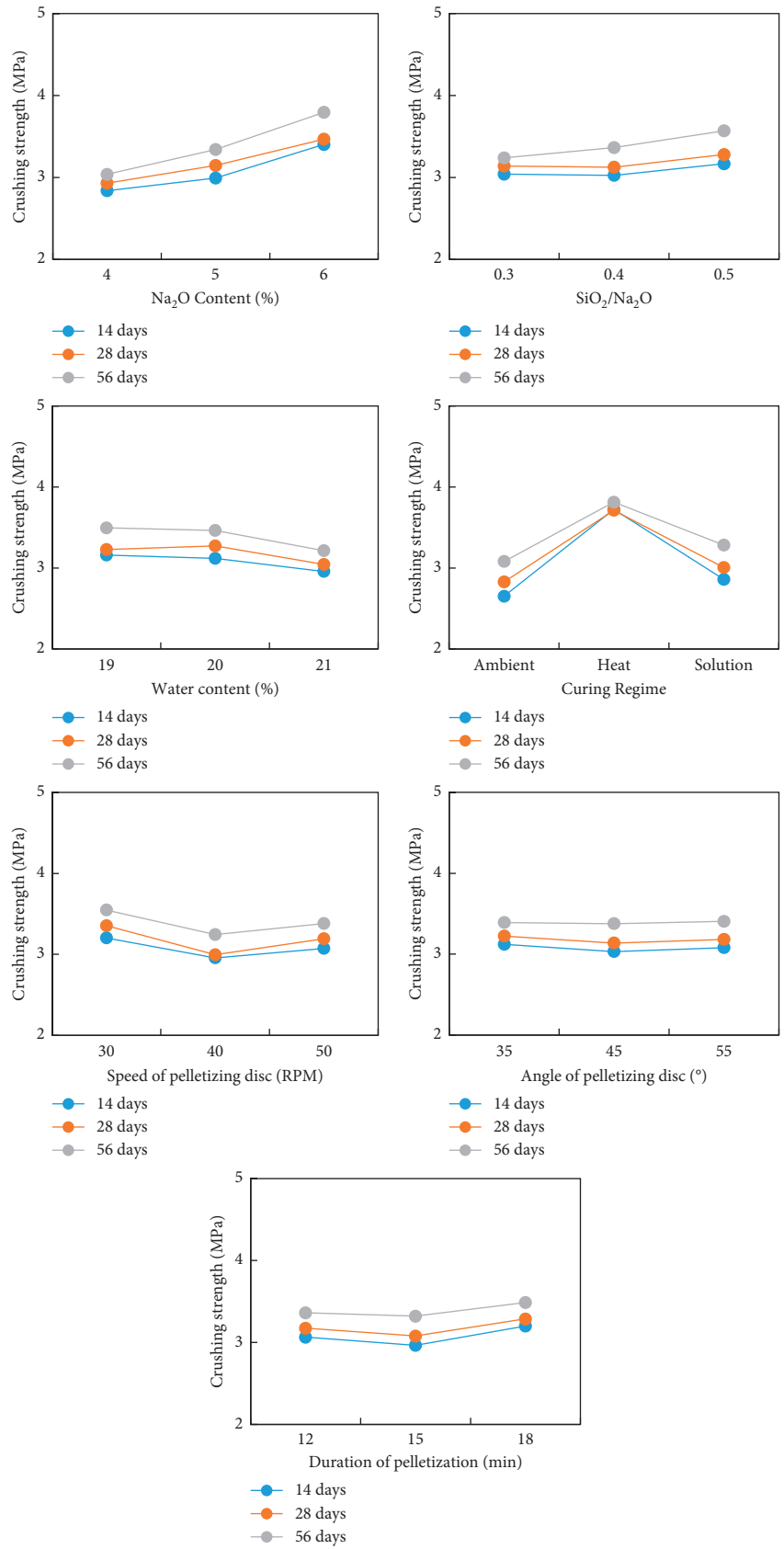
FIGURE 6: Response index relationship for crushing strength of individual pellets with combined factors.

Figure 7: Response index relationship for water absorption with combined factors.

TABLE 5: Grey relational generation, grey relational coefficients, and grey relational grade for different trial runs at 14 days.

Runs	Grey relational generations				Δ_{0i}				Grey relational coefficients				Grey relational grade (γi)
	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	
TMC 1	0.000	0.036	0.100	0.129	1.000	0.964	0.900	0.871	0.333	0.342	0.357	0.365	0.349
TMC 2	0.516	0.514	0.508	0.235	0.484	0.486	0.492	0.765	0.508	0.507	0.504	0.395	0.479
TMC 3	0.236	0.197	0.198	0.412	0.764	0.803	0.802	0.588	0.396	0.384	0.384	0.459	0.406
TMC 4	0.164	0.032	0.000	0.329	0.836	0.968	1.000	0.671	0.374	0.341	0.333	0.427	0.369
TMC 5	0.695	0.538	0.321	0.165	0.305	0.462	0.679	0.835	0.621	0.520	0.424	0.374	0.485
TMC 6	0.390	0.321	0.136	0.388	0.610	0.679	0.864	0.612	0.450	0.424	0.367	0.450	0.423
TMC 7	0.072	0.000	0.060	0.588	0.928	1.000	0.940	0.412	0.350	0.333	0.347	0.548	0.395
TMC 8	0.478	0.442	0.345	0.200	0.522	0.558	0.655	0.800	0.489	0.472	0.433	0.385	0.445
TMC 9	0.129	0.149	0.169	0.729	0.871	0.851	0.831	0.271	0.365	0.370	0.376	0.649	0.440
TMC 10	0.346	0.245	0.079	0.588	0.654	0.755	0.921	0.412	0.433	0.398	0.352	0.548	0.433
TMC 11	0.179	0.052	0.097	0.353	0.821	0.948	0.903	0.647	0.379	0.345	0.356	0.436	0.379
TMC 12	0.689	0.586	0.612	0.153	0.311	0.414	0.388	0.847	0.616	0.547	0.563	0.371	0.524
TMC 13	0.094	0.213	0.174	0.600	0.906	0.787	0.826	0.400	0.356	0.388	0.377	0.556	0.419
TMC 14	0.138	0.036	0.129	0.471	0.862	0.964	0.871	0.529	0.367	0.342	0.365	0.486	0.390
TMC 15	0.519	0.510	0.429	0.000	0.481	0.490	0.571	1.000	0.510	0.505	0.467	0.333	0.454
TMC 16	0.336	0.522	0.204	0.647	0.664	0.478	0.796	0.353	0.430	0.511	0.386	0.586	0.478
TMC 17	0.239	0.209	0.160	0.376	0.761	0.791	0.840	0.624	0.397	0.387	0.373	0.445	0.401
TMC 18	0.519	0.470	0.483	0.247	0.481	0.530	0.517	0.753	0.510	0.485	0.491	0.399	0.471
TMC 19	0.717	0.795	0.597	0.329	0.283	0.205	0.403	0.671	0.639	0.709	0.554	0.427	0.582
TMC 20	0.396	0.165	0.177	0.612	0.604	0.835	0.823	0.388	0.453	0.374	0.378	0.563	0.442
TMC 21	0.305	0.261	0.166	0.447	0.695	0.739	0.834	0.553	0.418	0.404	0.375	0.475	0.418
TMC 22	1.000	1.000	0.605	0.388	0.000	0.000	0.395	0.612	1.000	1.000	0.559	0.450	0.752
TMC 23	0.594	0.333	0.496	0.824	0.406	0.667	0.504	0.176	0.552	0.429	0.498	0.739	0.554
TMC 24	0.381	0.205	0.194	0.788	0.619	0.795	0.806	0.212	0.447	0.386	0.383	0.702	0.479
TMC 25	0.925	0.871	1.000	0.235	0.075	0.129	0.000	0.765	0.869	0.796	1.000	0.395	0.765
TMC 26	0.462	0.305	0.273	1.000	0.538	0.695	0.727	0.000	0.482	0.418	0.407	1.000	0.577
TMC 27	0.355	0.036	0.285	0.753	0.645	0.964	0.715	0.247	0.437	0.342	0.412	0.669	0.465

TABLE 6: Grey relational generation, grey relational coefficients, and grey relational grade for different trial runs at 28 days.

Runs	Grey relational generations				Δ_{0i}				Grey relational coefficients				Grey relational grade (γ_i)
	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	
TMC 1	0.132	0.058	0.092	0.167	0.868	0.942	0.908	0.833	0.366	0.347	0.355	0.375	0.361
TMC 2	0.431	0.602	0.475	0.117	0.569	0.398	0.525	0.883	0.468	0.557	0.488	0.362	0.468
TMC 3	0.268	0.394	0.239	0.333	0.732	0.606	0.761	0.667	0.406	0.452	0.397	0.429	0.421
TMC 4	0.000	0.149	0.000	0.222	1.000	0.851	1.000	0.778	0.333	0.370	0.333	0.391	0.357
TMC 5	0.637	0.593	0.309	0.000	0.363	0.407	0.691	1.000	0.580	0.551	0.420	0.333	0.471
TMC 6	0.298	0.473	0.088	0.346	0.702	0.527	0.912	0.654	0.416	0.487	0.354	0.433	0.423
TMC 7	0.275	0.000	0.095	0.407	0.725	1.000	0.905	0.593	0.408	0.333	0.356	0.458	0.389
TMC 8	0.420	0.485	0.283	0.080	0.580	0.515	0.717	0.920	0.463	0.493	0.411	0.352	0.430
TMC 9	0.108	0.382	0.176	0.525	0.892	0.618	0.824	0.475	0.359	0.447	0.378	0.513	0.424
TMC 10	0.403	0.340	0.114	0.630	0.597	0.660	0.886	0.370	0.456	0.431	0.361	0.574	0.456
TMC 11	0.098	0.041	0.266	0.469	0.902	0.959	0.734	0.531	0.357	0.343	0.405	0.485	0.397
TMC 12	0.671	0.618	0.554	0.154	0.329	0.382	0.446	0.846	0.603	0.567	0.528	0.372	0.518
TMC 13	0.183	0.485	0.258	0.451	0.817	0.515	0.742	0.549	0.380	0.493	0.402	0.476	0.438
TMC 14	0.251	0.100	0.033	0.346	0.749	0.900	0.967	0.654	0.400	0.357	0.341	0.433	0.383
TMC 15	0.468	0.622	0.540	0.006	0.532	0.378	0.460	0.994	0.484	0.570	0.521	0.335	0.477
TMC 16	0.498	0.602	0.190	0.525	0.502	0.398	0.810	0.475	0.499	0.557	0.382	0.513	0.488
TMC 17	0.149	0.207	0.193	0.290	0.851	0.793	0.807	0.710	0.370	0.387	0.383	0.413	0.388
TMC 18	0.414	0.618	0.423	0.136	0.586	0.382	0.577	0.864	0.460	0.567	0.464	0.367	0.464
TMC 19	0.569	0.813	0.499	0.160	0.431	0.187	0.501	0.840	0.537	0.728	0.499	0.373	0.535
TMC 20	0.424	0.515	0.115	0.660	0.576	0.485	0.885	0.340	0.465	0.507	0.361	0.596	0.482
TMC 21	0.386	0.573	0.188	0.432	0.614	0.427	0.812	0.568	0.449	0.539	0.381	0.468	0.459
TMC 22	1.000	0.946	0.613	0.358	0.000	0.054	0.387	0.642	1.000	0.903	0.564	0.438	0.726
TMC 23	0.786	0.701	0.495	0.920	0.214	0.299	0.505	0.080	0.701	0.626	0.497	0.862	0.671
TMC 24	0.600	0.295	0.153	0.654	0.400	0.705	0.847	0.346	0.556	0.415	0.371	0.591	0.483
TMC 25	0.851	1.000	1.000	0.235	0.149	0.000	0.000	0.765	0.770	1.000	1.000	0.395	0.791
TMC 26	0.736	0.432	0.364	1.000	0.264	0.568	0.636	0.000	0.654	0.468	0.440	1.000	0.641
TMC 27	0.525	0.299	0.351	0.821	0.475	0.701	0.649	0.179	0.513	0.416	0.435	0.736	0.525

TABLE 7: Grey relational generation, grey relational coefficients, and grey relational grade for different trial runs at 56 days.

Runs	Grey relational generations				Δ_{0i}				Grey relational coefficients				Grey relational grade (γi)
	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	Aggregate impact value	Aggregate crushing value	Crushing strength of pellets	Water absorption	
TMC 1	0.106	0.032	0.000	0.163	0.894	0.968	1.000	0.837	0.359	0.341	0.333	0.374	0.352
TMC 2	0.413	0.498	0.511	0.068	0.587	0.502	0.489	0.932	0.460	0.499	0.506	0.349	0.453
TMC 3	0.364	0.330	0.155	0.524	0.636	0.670	0.845	0.476	0.440	0.427	0.372	0.512	0.438
TMC 4	0.099	0.045	0.055	0.143	0.901	0.955	0.945	0.857	0.357	0.344	0.346	0.368	0.354
TMC 5	0.650	0.575	0.202	0.000	0.350	0.425	0.798	1.000	0.588	0.540	0.385	0.333	0.462
TMC 6	0.459	0.516	0.147	0.537	0.541	0.484	0.853	0.463	0.480	0.508	0.370	0.519	0.469
TMC 7	0.173	0.000	0.048	0.469	0.827	1.000	0.952	0.531	0.377	0.333	0.344	0.485	0.385
TMC 8	0.470	0.543	0.242	0.129	0.530	0.457	0.758	0.871	0.485	0.522	0.397	0.365	0.442
TMC 9	0.205	0.385	0.229	0.701	0.795	0.615	0.771	0.299	0.386	0.448	0.394	0.626	0.463
TMC 10	0.438	0.271	0.106	0.735	0.562	0.729	0.894	0.265	0.471	0.407	0.359	0.653	0.472
TMC 11	0.152	0.109	0.167	0.367	0.848	0.891	0.833	0.633	0.371	0.359	0.375	0.441	0.387
TMC 12	0.657	0.525	0.526	0.129	0.343	0.475	0.474	0.871	0.593	0.513	0.513	0.365	0.496
TMC 13	0.191	0.448	0.266	0.524	0.809	0.552	0.734	0.476	0.382	0.475	0.405	0.512	0.444
TMC 14	0.304	0.122	0.150	0.313	0.696	0.878	0.850	0.687	0.418	0.363	0.370	0.421	0.393
TMC 15	0.470	0.566	0.430	0.054	0.530	0.434	0.570	0.946	0.485	0.535	0.467	0.346	0.458
TMC 16	0.611	0.787	0.310	0.578	0.389	0.213	0.690	0.422	0.563	0.702	0.420	0.542	0.557
TMC 17	0.000	0.308	0.302	0.265	1.000	0.692	0.698	0.735	0.333	0.419	0.417	0.405	0.394
TMC 18	0.449	0.525	0.467	0.177	0.551	0.475	0.533	0.823	0.476	0.513	0.484	0.378	0.463
TMC 19	0.611	0.842	0.479	0.095	0.389	0.158	0.521	0.905	0.563	0.759	0.490	0.356	0.542
TMC 20	0.562	0.371	0.220	0.810	0.438	0.629	0.780	0.190	0.533	0.443	0.390	0.724	0.523
TMC 21	0.378	0.348	0.183	0.401	0.622	0.652	0.817	0.599	0.446	0.434	0.380	0.455	0.429
TMC 22	1.000	1.000	0.653	0.449	0.000	0.000	0.347	0.551	1.000	1.000	0.590	0.476	0.766
TMC 23	0.724	0.729	0.606	0.939	0.276	0.271	0.394	0.061	0.645	0.648	0.559	0.891	0.686
TMC 24	0.576	0.407	0.307	0.707	0.424	0.593	0.693	0.293	0.541	0.458	0.419	0.631	0.512
TMC 25	0.880	0.977	1.000	0.320	0.120	0.023	0.000	0.680	0.806	0.957	1.000	0.424	0.797
TMC 26	0.845	0.611	0.462	1.000	0.155	0.389	0.538	0.000	0.763	0.562	0.482	1.000	0.702
TMC 27	0.604	0.321	0.529	0.864	0.396	0.679	0.471	0.136	0.558	0.424	0.515	0.786	0.571

TABLE 8: Grey relational grade with respect to combined factors at various ages.

Factor	Curing ages	Grey relational grade			Maximum value–minimum value	Rank
		Level 1	Level 2	Level 3		
Na$_2$O content (%)		0.421	0.439	0.559	0.138	2
SiO$_2$/Na$_2$O ratio		0.446	0.481	0.493	0.047	6
Water content (%)		0.486	0.491	0.443	0.048	4
Speed of pelletizing disc (RPM)	14 days	0.478	0.452	0.489	0.037	7
Angle of pelletizing disc (°)		0.505	0.461	0.453	0.051	3
Duration of pelletization (min)		0.460	0.456	0.504	0.048	4
Curing regime		0.405	0.551	0.464	0.146	1
Na$_2$O content (%)		0.416	0.445	0.590	0.175	1
SiO$_2$/Na$_2$O ratio		0.455	0.492	0.504	0.049	4
Water content (%)		0.497	0.509	0.446	0.062	3
Speed of pelletizing disc (RPM)	28 days	0.501	0.452	0.499	0.049	4
Angle of pelletizing disc (°)		0.504	0.481	0.466	0.038	6
Duration of pelletization (min)		0.484	0.469	0.499	0.030	7
Curing regime		0.416	0.542	0.494	0.126	2
Na$_2$O content (%)		0.424	0.451	0.614	0.190	1
SiO$_2$/Na$_2$O ratio		0.455	0.505	0.530	0.076	3
Water content (%)		0.513	0.523	0.453	0.070	4
Speed of pelletizing disc (RPM)	56 days	0.512	0.466	0.512	0.047	5
Angle of pelletizing disc (°)		0.519	0.493	0.478	0.041	6
Duration of pelletization (min)		0.491	0.482	0.517	0.034	7
Curing regime		0.420	0.542	0.528	0.123	2

coefficients (GRC) plays a vital role in grey relational analysis, which correlates the desired and actual obtained results. The GRC was calculated by using equation (4) and computed GRC are presented in Tables 5–7 with respect to different trial runs at different curing ages.

$$\xi_i(k) = \frac{\Delta_{\min} + \psi \Delta_{\max}}{\Delta_{0i}(k) + \psi \Delta_{\max}}, \qquad (4)$$

where $\Delta_{0i} = \|x_0(k) - x_i(k)\|$ is the difference of the absolute value between $x_0(k)$ and $x_i(k)$ and $\Delta_{\max}$ and $\Delta_{\min}$ = maximum and minimum values of the absolute differences of all comparing sequences, respectively. ψ is a distinguishing coefficient $(0 \le \psi \le 1)$ and in the present study, $\psi = 0.5$ is taken.

The Grey relational grade (GRG) γ_i is calculated using equation (5) and presented in Tables 5–7 at various curing ages. If the Grey relational grade is higher it signifies a stronger relational degree between the investigated trial run and the ideal sequence.

$$\gamma_i = \frac{1}{n} \sum_{k=1}^{n} AP(k)\xi_i(k), \qquad (5)$$

where n = number of process responses.

The GRG was computed for individual factors with respect to combined factors and the values of GRG are presented in Table 8. From Table 8, it is observed that the highest GRG among the geopolymerisation factors is 6% of Na$_2$O content (level 3) followed by curing regime–heat curing (level 2) and 0.5 of SiO$_2$/Na$_2$O ratio (level 3) and among the pelletization factors the highest GRG is observed for angle of pelletization disc (level 1) and followed by 18 minutes of duration of pelletization (level 3) and speed of pelletization disc at 50 RPM (level 3). Whereas water content is the important factor which plays important

role in geopolymerisation and pelletization process, it is observed that higher GRG is observed for 20% of water content (level 2).

The level of significance of each individual factor is estimated through difference of maximum and minimum values. It is ranked according to the higher differences in the values of Grey relational grade and is presented in Table 8. From the table, based on the rank given in the grey relational analysis for the geopolymerisation factors, it clearly indicates that the curing regime and higher Na$_2$O content have major role in the aggregates production at early ages. However, the higher Na$_2$O content plays significant role in improving the engineering properties of the produced aggregates. Further, it is observed that curing regime rank is changed since solution curing of produced aggregates has improved overall engineering properties of aggregates at later ages. It is clear that, in the fly ash aggregate production with alkaline solution, Na$_2$O content, curing regime, and SiO$_2$/Na$_2$O ratio play major role in the rate of geopolymerisation of fly ash. Further, the GRG for factors of pelletization such as speed of pelletizing disc, angle of pelletizing disc, and duration of pelletization has relatively less influence as compared to the factors of geopolymerisation such as Na$_2$O content, SiO$_2$/Na$_2$O ratio, water content, and curing regime in the production of artificial aggregates. However, water content plays a prominent and dual role in geopolymerisation process and pelletization which acts as a medium in the process.

5. Discussion

The response indices of individual properties of aggregates from Figures 4 to 7 and combined effect in the grey relational analysis from Table 8 clearly show that higher Na$_2$O content, higher SiO$_2$/Na$_2$O ratio, and heat curing have significant

influence on the properties of APFA aggregates due to higher rate of geopolymerisation [15, 24]. Whereas the heat curing of aggregates increased the water absorption in the aggregates, solution curing has significantly improved water absorption in the aggregates which can be observed in the response indices in Figure 7, which corresponds to the higher degree of geopolymerisation of fly ash particles on the surface of the aggregates [10, 15, 24]. On the other hand, response indices of individual aggregates properties with different pelletization factors observed in Figures 4 to 7 and grey relational analysis from Table 8 clearly showed that the influence of the pelletization factors on the aggregate properties is negligible.

Further, it is observed that water content played an important role in the production process and to attain the necessary engineering properties of APFA aggregates due to following reasons. Firstly, in production stage, as the water content increases from 19% to 21%, the efficiency of production improved and particle size distribution of produced aggregates has marginally shifted towards coarser side. It is also observed from response indices of individual properties of aggregates from Figures 4 to 7 and combined effect in the grey relational analysis from Table 8 that an increase in water content from 19% to 21% has marginal and adverse effect on the properties of the APFA aggregates. In the geopolymerisation process of APFA aggregates, this water content may be used in early surface hydrolysis of fly ash particles or insufficient water for surface hydrolysis of fly ash which lead to less contribution to mechanical strength [17, 47, 48].

6. Conclusions

From this experimental study, it can be concluded that water content in alkaline solution plays a major role in the production process of the artificial aggregates and also to their engineering properties. Water content of 20% by mass of fly ash was found to be essential for the suitable production of artificial aggregates. It is observed that engineering properties of produced aggregates are very much sensitive to the factors of the geopolymerisation such as Na_2O content, SiO_2/Na_2O ratio, and curing regime. The most vital factors such as Na_2O content and heat curing improved the engineering properties at an early age attributed to the faster rate of degree of polymerisation. However, solution curing of produced aggregates has improved overall engineering properties of aggregates at later ages due to continuous geopolymerisation process, since the aggregates have absorbed sodium silicate solution during the curing period and that contributes to engineering properties at later ages. Further, statistically designed experiments showed that geopolymerisation factors have significantly influenced the engineering properties of the APFA aggregates as compared to the pelletization factors.

Data Availability

The experimental data used to support the findings of this study are included within the article.

Conflicts of Interest

The authors declare that there are no conflicts of interest.

Authors' Contributions

The manuscript has been read and approved by all authors.

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