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Microwave Assisted Synthesis and Experimental Exploration of Geopolymer Lightweight Aggregate

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Abstract- This research is mainly focused on the production of artificial LWAs. The constituent materials are fly ash (FA) and silica fume (SF) as precursor, which are activated by combination of sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3). The handcrafted pellets are cured in microwave oven for five minutes. Physico-mechanical performance of all LWAs are evaluated and compared with natural and synthetic LWAs available in the literature. The experimental tests including morphology, density, porosity, water absorption, crushing strength and aggregate impact value are conducted. Results exhibit that density of formulated LWAs is 44% less compared to normal weight aggregates and lighter than many previously manufactured cold bonded and sintered LWAs. The evaluation of mechanical properties proved the suitability of microwave curing. Based on the preliminary findings, proposed technology appears to be a viable solution for the production of eco-friendly LWAs by allowing waste materials to be recycled and energy saving for the production of LWAs.

Keywords- Lightweight Aggregates; Geopolymerization; Microwave curing; Physical Properties; Mechanical Properties.

I. INTRODUCTION

By the time, environmental sustainability got to be a serious concern from the point of view of natural resources depletion and that of wastes generation. Utilization of industrial wastes as construction material may be a sound sustainable practice to dispose the waste causing environmental nuisance and conserve the available resources for future generations.

Concrete is abundantly used construction material worldwide because of its versatility and durability [1]. One of the most commonly encountered problem in concrete is its unit weight, which ultimately results in increased design load due to its self-weight, as a result translates into higher building cost. As concrete is mainly comprised of coarse aggregates (60-75%) which is responsible for its adequate strength and impart significant weight to concrete [2, 3]. Throughout the twenty-first century, the demand for the lightweight concrete has increased with the improvement of technology and the complicated constructions. Mostly, lightweight concrete is formulated by using LWAs. Naturally occurring LWAs include pumice, scoria, vermiculite, diatomite etc. and their densities range from 88-600 kg/m^3 [4, 5]. Construction industry is the large consumer of natural resources and their continuous usage threatens environmental sustainability. As far as the consumption of natural LWAs is concerned, its shortage in the growing infrastructure industry builds the need for artificial LWAs. Researchers have carried out extensive work on this area, trying for new alternative material for this deficiency in construction industry and in order to avoid aggregate scarcity for the present world, also to deal with declining availability of natural resources, which are getting overexploited [6-9]. Replacement to natural aggregates, artificial LWAs are encouraged through utilization of industrial waste which has a great role in terms of environmental impacts, economic considerations as well as better performance characteristics. Artificial LWA production from different waste streams has a promising future due to its positive influence on the environment, the community and the construction industry [10]. In the present scenario, the progress of industry and technology led to produce substantial quantity of waste materials globally which have a potential to be utilized as supplementary cementitious materials (SCMs). To minimize and recycle the large quantities of industrial and agricultural waste materials or by products (such as rice husk ash, fly ash, slag, silica fume, bagasse, stone sludge, brick dust) has been employed in manufacturing of artificial aggregates [8, 11, 12] as their use is a suitable way to minimize energy consumption and environmental impacts. The cost-effective production strategy for obtaining LWA using FA may be a real sustainable material for future construction.

Agglomeration and palletization process are

mechanisms that envisages the formulation of pallets from a powder material with more stable spherical balls. At the beginning both methods let the waste materials be palletized with other adhesive agents [13, 14]. Preliminary characterization is taken into consideration while selecting material for aggregate production. FA as a supplementary cementitious material (SCMs) has been reported as the most commonly used byproduct for the production of aggregates due to its particle size distribution and chemical composition [15]. Also the production of artificial aggregates is a great leap towards the FA disposal in large quantity. Since SCMs do not possess self-bonding capability so cementing and geopolymerization technique is used in concordance with agglomeration or palletization. Apart from cementing due to its inability in environmental sustainability, Alkaline activators have been widely reported to be used in geopolymerization. In addition to being economical, mixture of NaOH and Na_2SiO_3 is extensively used due to its wide availability [16, 17].
The curing of palletized pallets may be mainly accomplished by two distinct processes. The cold bonding process let the pallets be cured at ambient temperature or be stored in an enclosed space for 1,3 or 7 days. The sintering process let the pallets be cured in a rotary kiln at a temperature of 1000-1300^{o}C [18] and those assorted pallets became ready to use after they cool down. The later method consumes large amount of energy, causing disturbance in environmental sustainability, while former method is more economical due to minimum energy consumption.
From the advances in novel technologies for drying, microwave energy is becoming increasingly important heating, drying and curing energy source. Apart from the conventional curing techniques that relies on the conduction of heat from exterior to *interior, microwave curing which is based on* internal energy dissipation associated with the excitation of molecular dipoles in electromagnetic field, allows faster and more uniform heating, cleaner production and no generation of secondary wastes, high thermal efficiency as well as enhances production rate [19]. Microwaves have the ability to penetrate deep inside material in a short time and the water in a material easily achieves high temperature by volumetric heating which introduces foam in a mass by vaporization in a short while [20]. Microwave curing avoids high temperature demand and hazardous emissions as associated with sintering and it also avoids the time required for better bonding as mandatory in cold bonding process without compromising physico-mechanical properties of LWA [18]. So, it is important to analyze the efficiency of microwave curing as it would be advantageous towards the sustainable development.

II. EXPERIMENTAL METHODOLOGY

This study possesses a new methodology to produce LWAs through geopolymerization, incorporating two industrial byproducts. Microwave curing was adopted as a fast and efficient curing regime. After that, these ready-used aggregates were studied for their physical and mechanical behavior. Following sub-sections explain the experimental methodology of the proposed work.

A. Materials

In the production of LWAs, two materials were used as a precursor: FA and SF. FA was procured from the DG cement Pakistan and had a chemical composition presented in Table 1. Its composition resembled to Class-F FA confirming the specifications according to ASTM C618 [21]. Chemical composition of SF is also presented in Table 1. A mixture of NaOH and Na_2SiO_3 was used as alkaline activators in geopolymerization process, which was purchased from Akbari Mandi (Lahore, Pakistan). NaOH flakes were used to prepare 10M NaOH solution. Just before handcrafting of aggregates, $NaHCO_3$ (baking soda) was used in aggregate paste as an accelerator and surface hardening agent [22].
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Table 1: Chemical composition of FA and SF used in this work

Constituent	Fly Ash	Silica Fume
CaO	9.02	0.27
SiO_2	56.34	93.65
Al_2O_3	23.08	0.28
MgO	1.7	0.25
Fe_2O_3	6.43	0.58
K_2O	0.56	0.49
Na_2O	0.28	0.02
CI (%)	0.025	3.62
LOI (%)	< 3	<5
Moisture Content (%)	<1	-

B. Specimen Preparation and Curing

The first part of this study encompasses the manufacturing process of coarse LWAs using FA and SF via geopolymerization process. Selection of mix proportions for the production of LWAs is based on previous research knowledge and is performed on the basis of hit and trial method. FA was selected as main constituent of LWAs and SF was consumed in this process of production as 10 % of total solid. FA and SF were used in percentages of 90% and 10% respectively and 1% of total solid material was replaced by $NaHCO_3$. FA was major precursor and secondary precursor SF was added to study its effect on aggregate strength. Mixture of two alkaline solutions, 10M NaOH and Na_2SiO_3 were used in amount of 35% of total weight of mix. The ratio of two solutions $NaOH/Na_2SiO_3$ and the ratio of alkaline activator to solid were 0.42 and 0.53 respectively. Calculated amounts of materials were dry mixed first for about 2-3 minutes and then mixture was further mixed for 5-6 minutes after adding solution. After formation of homogenous mixture, 1% of $NaHCO_3$ was added and mixed thoroughly. After that aggregates were moduled by hand at laboratory level ranging 12-16mm diameter. After formation, pallets were kept in laboratory for 2-3 hours at room temperature to make them strong enough so that they can bear handling stress. After that, pallets were studied under single curing regime that was microwave radiation curing. Aggregates/Pallets were placed in mud pot, cured in microwave for about 4-5 minutes. After curing, these ready-used aggregates were wrapped in plastic bags to avoid moisture penetration in order to perform several tests.

Table 2. Mixing proportions for the production of LWA

Material	Proportion (%)
Fly ash	89
Silica fume	10
$NaHCO_3$	1
Na_2SiO_3	70
NaOH	30
Liquid/solid	0.53

Mix proportion for the formulation of LWAs is given in Table 2. The pellets designation is explained as: first two alphabets tell the primary precursor, Fly ash (FA), after alphabet first two numerals tell the percentage of NaOH, next two numerals tell the percentage of Na_2SiO_3, and last two alphabets are for secondary precursor, silica fume (SF). The specimen identity is FA30-70SF.

C Testing

LWAs were tested for two main fractions that are its physical and mechanical characteristics. Physical properties were assessed by some visual examination like color, texture angularity and some experimental testing like density, water absorption, specific gravity and porosity. While mechanical evaluation was done by particle crushing strength test and aggregate impact value test. The schematic diagram of whole methodology is presented in Figure 1.

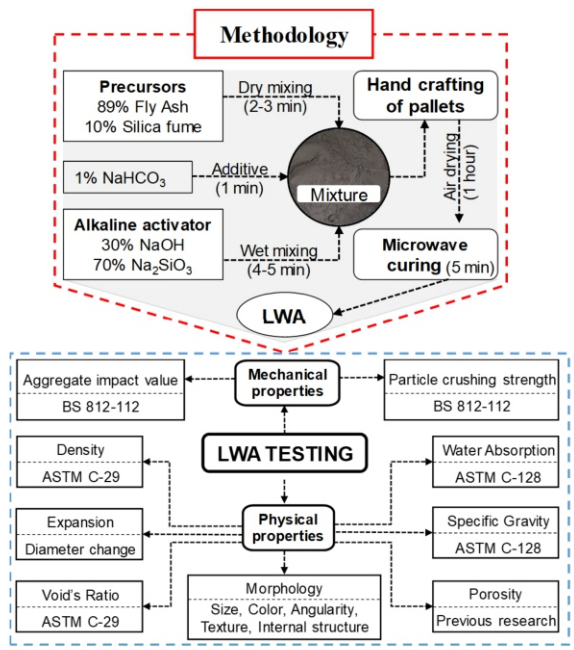

Figure 1: Summary of LWAs production and testing

III. Results and Discussions

Aggregate properties have direct impact on the performance of concrete, thus understanding and evaluation of these properties is very obligatory to make concrete with desired properties. The mechanical characterization of LWAs was made through the evaluation of particle crushing strength test and aggregate impact value test. The physical and mechanical properties of sample are summarized in Table 3. Moreover, results were also compared with natural LWAs and aggregates available in literature: cold bonded LWAs, Sintered LWAs.

A. Morphological Characteristics

Aggregate particles shape and texture directly influence the fresh properties of concrete more than the properties of hardened concrete [23]. Rounded shape and surface smoothness of the aggregate enhanced the viscosity and workability of concrete. Comparatively aggregates with angular shape and rough texture require more water for concrete production [4]. Pallets produced in this study were spherical in shape and had shiny surface before curing. After curing they were remained spherical and smooth textured but, on the micro-scale, surface was slightly rough with small open pores. Aggregate shape has a major impact on particle packing and interlocking within the mix. Its surface texture can influence frictional properties during fresh state and consequently harshness of the concrete mix.

Color of artificially produced aggregates majorly depends on the precursor used for their production. In this study it was observed that their color was dark grey before curing and after curing a little color change was noted:light grey with internal grey core.

Grading of aggregate is also a very important factor to consider because it determines the paste requirements such as water requirement and cement content for concrete. Various sizes of aggregates were produced in this study. Average particle size to be observed was 13.2±0.1mm, the smallest size was 11±0.1mm and largest size was of 15±0.1mm. All produced aggregates were coarse aggregates as they retained on sieve # 4.

Microscopic analysis showed the internal porous structure of aggregates. The structure of the pores was observed as well distributed. They have a relatively uniform system of pores. Pores close to surface are readily permeable and fill rapidly after water immersion while inner pores fill in relatively slow.

B. Expansion

Microwave curing introduces pores on the outer skeleton and inner structure of aggregates, due to which aggregate volume was increased. It was observed that expansion of FA30-70SF was 15.20%. Na_2SiO_3 was used as expansive agent to create pores within the aggregate paste since microwave heating induces the silicate group to react and expansion continues until the gelling of silicate groups [20] which is retained upon cooling. LWAs produced from of shale and slate showed 59-68% expansion after bloating [24] while 8.8% expansion was recorded for basic oxygen furnace slag after autoclaving [25]. The expansion of mix depends upon curing method, also it is affected by the expansive agent used. In the view of literature, it may be noted that expansion from microwave radiations was better than autoclaving.

C. Bulk Density and Void's Percentage

Aggregate density is a decisive factor determining the unit weight of concrete and ultimately the dead load or self weight of concrete structures. Bulk density of normal weight aggregate varies from 1200 to 1760 kg/m^3. European standard BS EN 13055 [26] specifies aggregates having loose bulk densities not exceeding 1200 kg/m^3 as LWAs. Depending upon the size of aggregates loose bulk density of 880-1120 kg/m^3 is permissible for the production of structural lightweight concrete according to ASTM C330 [27]. Density of sample was measured after 5 minutes of microwave curing and it was observed that it was less than benchmark (880 kg/m^3) given by ACI 213R-03 [28], that is 711 kg/m^3 which verified its applicability as LWA.

It can be observed from Figure 2 that the density of sample was higher than natural LWAs but lower than that of many cold bonded and sintered LWAs available in literature.

The percentage void value depends on the shape of aggregates, size of aggregates and degree of

Table 3: Physical and Mechanical properties of manufactured LWA

Properties of LWA	Units	Standards	Threshold value	Observed value	Standard deviation (%)
Loose bulk density	kg/m^3	ASTM C330	880	710	19
Porosity	%	Literature	Up to 67	27.07	59
Expansion	%	Literature	8.8-68	15.20	-72, +77
Water absorption	%	ACI 213R	>25	17.95	28
Specific gravity (OD)	---	Literature	1.29-1.85	1.8	-39, +2.7
Particle crushing strength	MPa	Literature	2.03-12.00	3.34-3.54	-69, +71
Aggregate impact value	%	BS-882	<35	10.03	71%

Table 4: Relative density of different LWAs

Relative Density	FA30-70SF	Sintered LWAs			Cold Bonded LWAs						Natural LWAs		
	FA-SF	FA+B	FA+GP	FA+C	B	FA+B	FA+C	FA	FA+C	FA+C	pumice	scoria	Diatomite
		[9]	[32]	[33]	[34]	[9]	[14]	[32]	[14]	[14]	[4, 35]	[35, 36]	[37]
SSD	1.80	1.57	1.60	-	-	1.85	1.65	1.63	1.69	1.75	-	-	-
OD	1.50	1.56	1.59	1.40	1.63	1.62	1.23	1.3	1.29	1.44	1.14	1.10	1.32

compaction (how densely the aggregates are packed). The value of percentage voids for sample was 5.77%. The compaction was done on microlevel by hands so, there might be a chance of deviation in force exerted during compaction. The larger pallets are less compacted in their outer layers with subsequent larger voids. Angularity enhances void content while greater amount of well graded aggregates reduces void content.

D. Water Absorption

The water absorption test was carried out for sample having different sizes, ranges from 11mm to 17mm, and water absorption values were calculated after 24h immersion in water. The observations are presented in Figure 3 and compared with natural LWAs, previously manufactured sintered and cold bonded LWAs. It was observed that sample have water absorption of 17.95% which is less than natural LWA due to lesser porosity than natural LWA.

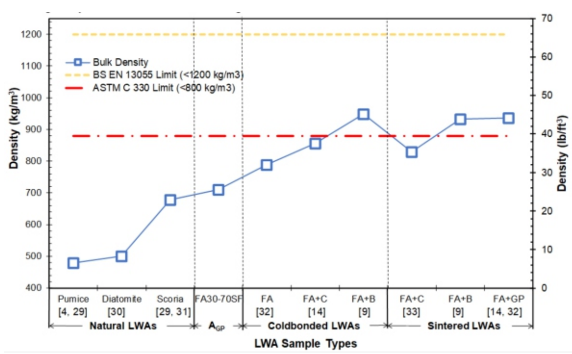

Figure 2: Bulk Density of Different LWAs

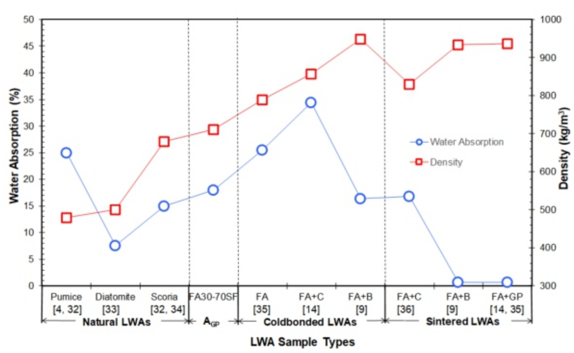

Figure 3: Relationship of density and water absorption of Different LWAs

Figure 3 depicts that formulated aggregates have water absorption within normal range for LWA (5-25%) according to ACI-213R [28]. However, most of the commercial artificial LWA exhibits water absorption around 18% [29]. Due to their cellular structure, LWAs absorb more water than their ordinary aggregate counterparts. Based upon a 24-hour absorption test conducted in accordance with the procedures of ASTM C 127 [30] and ASTM C 128 [31], structural-grade LWAs will absorb from 5% to more than 25% moisture by mass of dry aggregate. By contrast, ordinary aggregates generally absorb less than 2 % of moisture. Rate of absorption is unique to each LWA, and is dependent on the characteristics of pore size, continuity, and distribution, particularly for those pores close to the surface [28].

More water absorption of LWAs is associated to lesser density of LWAs, which is an indication of porous microstructure. From Figure 3 it is concluded that as the density of LWAs increasing, water absorption trend is decreasing because lesser density means aggregates are more porous and porous aggregate absorb more water than less porous (low density) aggregates.

E. Specific Gravity

Relative density of LWA sample is calculated and presented in Table 4. Relative density; Oven dry and saturated surface dry are 1.5 and 1.8 respectively, 20-45% less than that of normal weight aggregate. Normal weight aggregates have relative densities range from 2.40-2.90, which means that these aggregates are 2.40-2.90 times heavier than water. According to ACI-213R [28], the relative density of LWAs is 1/3 to 2/3 of normal weight aggregates. The LWAs manufactured in this study fulfill the requirements of AC1-213R. However, natural LWAs are much lighter. Formulated LWAs exhibit relative density in between previously manufactured sintered LWAs ranging from 1.40-1.59 and cold bonded LWAs ranging from 1.29-1.85. Less specific gravity of aggregates is associated to lesser density of aggregates, which is attributed to higher porosity. Although higher specific gravity of aggregate reflects its high strength, but it is impossible to assess its suitability on this basis alone without considering several mechanical properties.

F. Aggregate Porosity

Aggregate total porosity test assists to determine percentage of total pores in aggregate. Total porosity, particle density true density of sample were determined and observed values are presented in Table 5.

Table 5: Observation for aggregate porosity test

Property	Value
Particle Density (kg/m^3)	1473
True density (kg/m^3)	2070
True porosity (%)	27.07

Na_2SiO_3 helps to introduce closed pores in aggregate internal structure ultimately due to which true or total porosity increases. Natural LWAs exhibit higher porosity percentages than synthetic aggregates. Table 6 presents the porosity percentage of natural LWAs, cold bonded and sintered LWAs available in literature. Maximum total porosity of LWAs can be up to 67% [38]. So, the porosity of formulated LWAs was within the normal range. Moreover, porosity of aggregate has inverse relationship with density, because with the increased porosity more closed pores create in aggregate which ultimately leads to increase the volume and reduce the density. These all findings endorse the theory that increase in porosity leads to reduced density.

Table 6: Porosity of different LWAs

Type of Aggregate	Binder	Porosity (%)
Microwave	FA-SF	27.07
Sintering	FA+B [32]	8.4
	FA+GP [32]	6.2
Cold Bonded	FA [32]	31.1
Natural LWAs	Pumice [4, 35]	59.46
	Scoria [35, 36]	40.04

G. Particle Crushing Strength

The particle crushing strength test was conducted on a range of aggregates (11mm - 15mm) for sample that shows decrease in the value of crushing strength with the increase in particle size. However, discrepancy in preceding trend for some particles was there due to the non-uniformity of surface. The highest particle crushing strength of 4.54 MPa was recorded as shown in Table 7.

Table 7: Crushing strength test results

Pellet Size	Strength (MPa)
11	3.34
12	4.54
13	3.66
14	3.58
15	3.86

By comparing the samples and correlating the relative density of samples it was observed that with the decrease in density, compressive strength of aggregates also reduced. It was concluded that samples containing FA and SF exhibit good compressive strength as SF is very reactive pozzolanic material due to its extreme fineness and very high amorphous silicon dioxide content. The highest compressive strength of FA30-70SF due to the inclusion of SF may be related to the packing effect of SF which act as a filler in voids among FA. Moreover, fine nature of SF caused prominent improvements in pore size distribution that leads to good compressive strength [39].

In comparison with literature, the particle crushing strength of sample lies in the range of 2.03-12.00 MPa with particle sizes of 10-20mm [8, 17]. LWA produced from sintering have high particle crushing strength as compared to cold bonding, varying from 3.70-20.62 MPa. While sample shows better strength tha natural LWA, pumice having particle crushing strength of 1.49-1.96MPa [40].

H. Aggregate Impact Value

The aggregate impact value test was carried out for sample having particle sizes that retain on 0.36mm opening and the results for aggregate impact value were came out to be 10.03%. The lower the impact value is, better will be the resistance of aggregates against impact loads. For normal weight aggregates, the impact values below 10% are considered as strong while values above 35% are normally considered as weak aggregates for construction applications [41]. While BS 882-1992 [42] describes the maximum impact value as 25% when aggregate is to be used in heavy duty floors, 30% when aggregate is to be used in concrete for wearing surfaces and 45% for other concrete applications. From standard threshold values, manufactured LWAs observed to be strong enough to use in concrete. Sample exhibited lower impact value as comparatively stronger to sudden impact compared to LWAs formulated in previous research having aggregate impact value in the range of 22.10-35.70% [11, 43]. It was observed that the sample was also stronger than natural LWA having aggregate impact value of 15.63% [44] and 33.2% [36] and this was because of its greater density than natural LWAs. It may be concluded from Figure 3 that aggregates with higher values of density and lower values of water absorption shows good strength as they impart lower impact values and vice versa.

IV. CONCLUSIONS

In this work, LWAs were synthesized from abundantly available waste materials (FA and SF) through geopolymerization technique and cured using microwave radiations. Based on the results of this experimental investigation, several conclusions were extracted and summarized as follows:

1. Microwave radiations provide fast route to adequate curing of aggregates. It also maintains a strategic distance to high temperature and time demand as required in other methods such as sintering, autoclaving and cold bonding.
2. After curing, LWA sample showed 14.17% expansion due to the addition of expansive agent (Na_2SiO_3) by weight of 70% of total liquid added.
3. Density of LWAs was less than threshold (880 kg/m^3) as recommended by ACI 213-R. Similarly, water absorption (17.95%) was also within the normal range of 25% by ACI and commercially available artificial LWAs.
4. The specific gravity in surface saturated dry

condition is 1/3 of normal weight aggregates which also shows the absence of deleterious materials and lumps.

5. Total porosity of LWAs is found out to be 27.07% which is less than threshold (67%) and also lesser bulk density and relative density of aggregates indicate its porous microstructure.
6. LWAs showed good performance in terms of mechanical behavior. Particle crushing strength was observed to be 3.34-4.54 MPa, which were within the acceptable range as in previous researches.

In this study, FA and SF, as waste material can be utilized to successfully produce LWAs by microwave curing technique. This product possesses well engineering properties and can be used in concrete to achieve sustainability. It is worthy to note that this technology may offer assistance to energy saving and resource conservation. Moreover, finite element analysis is recommended for in depth study of properties of LWA and also to evaluate the influence of microwave curing.

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