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Review paper

BIOMASS ASH IN ALKALI-ACTIVATED MATERIALS TECHNOLOGY - A SCOPING REVIEW

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Abstract

As the construction industry moves toward decarbonization, there is a growing focus on developing alternative binders with a lower carbon footprint to replace Portland cement. Alkali-activated materials (AAMs) have emerged as one of the promising solutions because of the lower CO_2 emissions from their production and comparable performance. The main components of AAMs are amorphous aluminosilicate-rich precursors that react with alkali activators to form a solid binding matrix. Raw materials generally used as precursors in AAMs are industrial by-products such as slag, coal combustion fly ash, or metakaolin. The most used activators to reach the desired performance of the binder are alkali hydroxides and alkali silicates. However, conventional alkali activators are chemicals characterized by a negative environmental impact and high cost, which limits the wide-scale application of AAMs in the construction sector. Replacing conventional chemical activators with alternative ones derived from waste materials is a promising solution to enhance the sustainability of AAMs while promoting their broader application. This paper presents a targeted scoping review on the use of different biomass ashes instead of chemical activators in the AAMs technology. The results showed that different biomass ashes can be used not only as a source of alkalis and silicates but also as a partial replacement for commonly used precursors.

Key words: *alkali-activated materials, biomass ash, alternative activator, decarbonization*

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1. INTRODUCTION

Alkali-activated materials (AAMs) are binders based on aluminosilicate-rich precursors and alkaline activators. Blast furnace slag (BFS), coal combustion fly-ash (FA) and metakaolin are the most widely used precursors, while alkali hydroxide, silicates, carbonates and sulphates can be used as activators. Depending on the chemical composition of the precursors the main reaction products are calcium-aluminosilicate hydrate type gel (C-A-S-H) and three-dimensional alkali-aluminosilicate hydrate type gel (N-A-S-H). AAMs with predominant N-A-S-H gel in the microstructure are usually referred to as geopolymers [1,2,3].

The most used activators are hydroxides and silicates (commercially known as “water glass”), sodium or potassium based. Due to their lower cost, sodium activators have broader application [1]. The alkalis in AAM technology provide high pH and initiate the formation of reaction products, promoting early strength development. Silicates contribute to the density of the matrix and high 28-day and long-term compressive strength of concrete. Furthermore, they enhance workability if applied in adequate amount [4,5].

Although AAMs can have lower CO_2 footprint than Portland cement binder, while keeping the satisfactory mechanical and durability performance, the energy-intense production of chemical activators significantly contribute to the carbon footprint of AAMs, along with other environmental challenges [6,7,8]. Therefore, in the past decade, research has focused on finding alternative sources of alkalis and silicon on alternative activators. Silica fume (SF) was reported as a suitable silica source in AAM technology for slag-based [6], fly ash [9] and metakaolin-slag blended AAMs [4]. Waste glass (WG) was also used as an alkali silicate activator for BFS [10] or FA and BFS-FA blends [11], metakaolin [12] and even as precursor [8].

Besides industrial by-products such as SF and WG, plant biomass ashes are reported in literature as a potential alternative activators or precursors, due to their high content of silicon, potassium and calcium, depending on the type of crop [13]. Biomass ashes are a waste material, resulting from the combustion of biomass ash as a renewable energy source. Worldwide, different crops residues are used as fuel, depending on the availability [8]. Since the biomass ashes are usually landfilled, the strategy to valorize it through AAM technology and address the environmental challenges of landfilling have become an interesting research topic.

This scoping review aims to present possibilities of using different plant biomass ashes in AAM technology. Due to the large number of variables, the data were structured according to the type of precursor and biomass ash used, accompanied by compressive strength values, without delving into detailed mix design information.

2. METHODS

The scoping review was conducted by searching the Scopus database, with the following key words: “alkali-activated materials”, “biomass ash”, “alternative activator”, “waste valorization”, “agricultural biomass ash”, “alternative silica source”, ”alternative alkali source”. Additionally, some papers were selected by screening the references of relevant papers.

The inclusion/exclusion criteria are based on the used precursor, alternative activator type and tested properties. Only papers on AAM based on BFS, FA, metakaolin and their blends were included. Papers addressing alternative alkali and silica activators and precursors

based on biomass ash were included, while papers on industrial by-products as alternative activators were excluded. Given the wide range of varying parameters reported in the literature (e.g., water-to-binder ratio, silica modulus, precursor-to-activator ratio, etc.), the present scoping review focuses on sample type (paste, mortar, or concrete), compressive strength, and curing conditions, with comments on activator preparation.

3. RESULTS

The summarized results are presented in the scheme in Figure 1. The data is structured to provide general information on the use of biomass ash. The type of precursor used with biomass ash is presented on the left (BFS, FA, metakaolin, blended systems). The data on attained compressive strength and curing regimes are given in detail in the subsections.

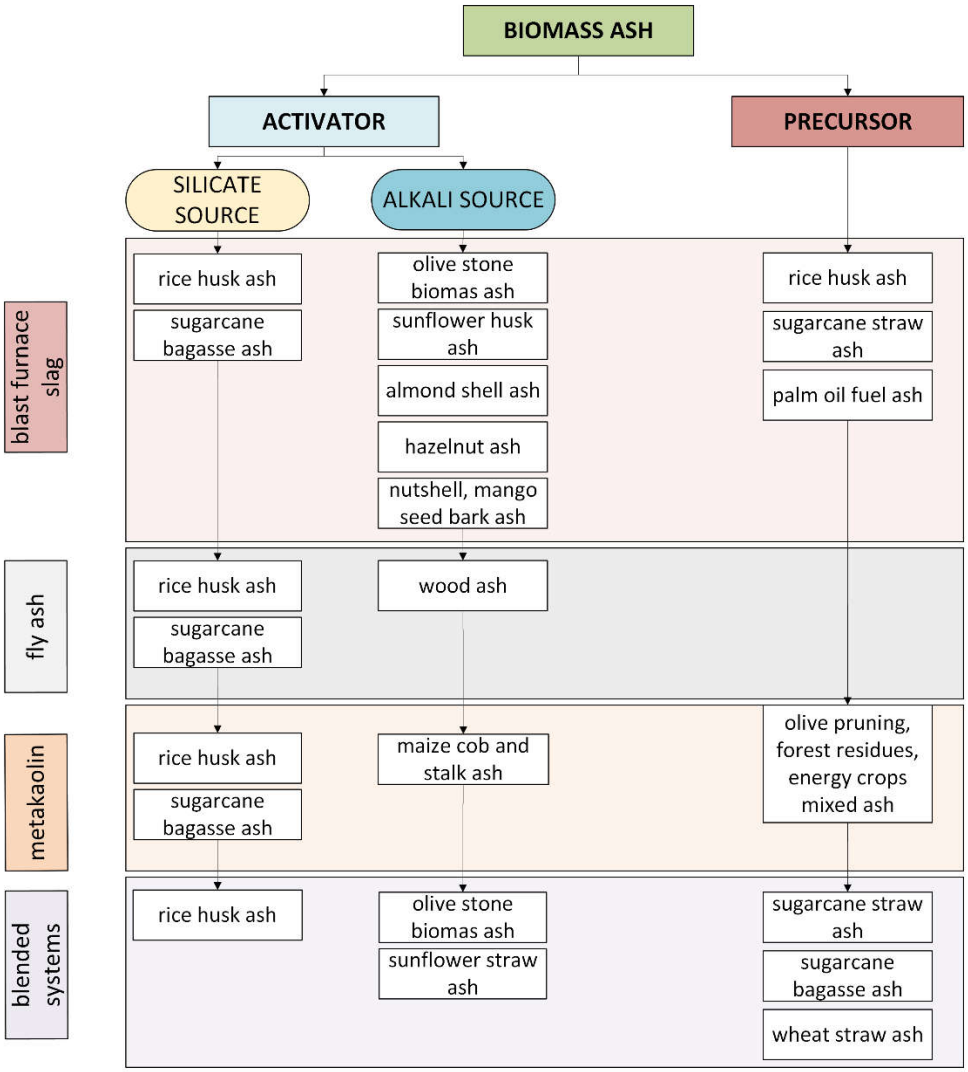

Figure 1. Schematic presentation of biomass ashes application in AAM technology

3.1 Biomass ashes as alternative activators

Biomass ashes investigated as alternative alkali silicate activators in AAM technology are rice husk ash (RHA) [4,14,15], sugarcane straw ash (SCSA) and sugarcane bagasse ash (SCBA), where the last two are also referred to as sugarcane biomass ash (SBA) [8]. These biomass ashes were used to activate BFS [14,16,17,12], FA [17,18], metakaolin [19,20,21] and blended systems: metakaolin-BFS [4], metakaolin-FA [22] and BFS-FA [17,23]. Silica sources usually must be treated to extract silica [8,9], by hydrothermal or thermochemical methods [6]. In the referenced research, RHA and SBA were mixed with NaOH solution to prepare an activator (two-part alkali-activation), usually 24h before mixing of AAMs, to ensure the dissolution of the silicon from the ashes. Some research investigated silicon extraction under elevated temperatures [18]. Paste or mortar samples were usually compared with reference mixes, activated with NaOH and commercial sodium silicate solution, or in one case, potassium silicate [19]. Although in some cases reference mixes did exhibit higher strengths, the overall performance of samples activated with alternative silica sources was comparable.

Most of the research on alternative alkali sources focused on BFS activation with: olive stone biomass ash (OBA) [24,25,26,27,28,29,30,31,32], sunflower husk ash (SHA) and sunflower straw ash (SSA) [29,30,31,33,32], almond [34] and hazelnut ashes [27,28]. Only a few papers were found on other precursors, including a combination of on BFS and RHA [15], metakaolin [35] and FA [36]. In all reviewed studies, a one-part alkali activation approach was applied (biomass ashes as activators are added as solids), while the two-part method was tested in only one case and was found to be less effective compared to the one-part alkali-activation [35].

Published research mostly investigated microstructure and compressive strength after 7 and/or 28 days of curing. Thermogravimetric analysis [4,16,37,24,15], Fourier Transform Infrared Spectroscopy [4,16,18,37,35], X-ray diffraction [22,37,24,15] were used to confirm the formation of C(N,K)-A-S-H and N-A-S-H gels in samples activated with alternative activators, depending on the chemical composition of biomass ashes and precursors. Some papers are also addressing the reactivity of component materials and workability of fresh mixes [32,38,28]. There is data on the excellent resistance against acid attack and lower capillary sorption of biomass ash activate concrete [39], however it is not the subject of this review.

3.1.1 Alternative silicate sources

Bernal et al. [14] tested RHA potential to act as silica source in activation of BFS pastes. Rise husks were burnt in the laboratory at 600˚C and ground to the desired particle size distribution. The obtained RHA had 68% of amorphous silica. RHA activated pastes showed higher 7 (>40 MPa) and 28-day compressive strength (>100MPa) than reference samples (75 MPa), activated with the commercial sodium silicate. Authors also investigated thermal stability, where RHA activated retained measurable compressive strength after exposure to 800˚C, while reference mix did not.

The use of RHA as a replacement for water glass was proven viable for BFS, FA and FA-BFS pastes [17]. BFS and blended systems were cured at 95% of relative humidity and ambient temperature, while FA pastes were cured at 80 ˚C, for 24 hours. Although the reference mix with conventional sodium silicate had compressive strength of 75 MPa after 7 days, RHA activated systems showed promising results at the same age, reaching

approximately 41 MPa for BFS-activated pastes, 49 MPa for FA and 39 MPa for blended system.

RHA and SCSA were investigated as activators for BFS mortars by Moraes et al. [16]. Two activator solutions were prepared by dissolving RHA and SCSA in NaOH. After curing at 65 °C for 3 days and at 20 °C for 28 days, the RHA-activated samples achieved compressive strengths of 49.7 MPa and 59.7 MPa, respectively, while the SCSA-activated samples reached 45.0 MPa and 54.9 MPa, respectively. However, neither of the alternative activators outperformed the reference samples, which achieved 62.9 MPa and 78.8 MPa under the same curing conditions.

Furthermore, FA was activated with RHA and SCSA to produce alkali-activated pastes in the work of Gomonsirisuk and Thavorniti [18]. After curing at ambient temperature for 7 days, samples synthesized using RHA exhibited the highest compressive strength of approximately 23 MPa, whereas the maximum strength achieved with SCBA was around 16 MPa.

Metakaolin-based alkali-activated pastes were successfully synthesized with RHA as silica source [40,37,19]. After 20 hours of curing at 72 °C and ambient curing up to 28 days, the achieved compressive strength is reported to be approximately 40MPa [19]. For the samples cured only at ambient conditions, 28-day compressive strength was reported to be 36.29 MPa [37] and 29.9 MPa which increased to 32.8 MPa after 56 days of curing [40]. Similar results were reported for activation of metakaolin with SCBA [20].

Blended metakaolin-BFS pastes activated with RHA was analyzed by Bernal et al. [4]. Authors varied BFS content and silicon-to-alumina ratio, which resulted in 7-day compressive strengths in range from 12 MPa to almost 60 MPa. Conducted curing regime was as follows: ambient conditions (25–30°C) for 24 h, then exposed to thermal curing at 60°C and >90% relative humidity for 24 hours. The samples were then stored at ambient temperature and 90% relative humidity until testing.

FA-BFS blended mortars were also activated with RHA in the work of Tong et al. [23]. RHA activated samples reached almost 60 MPa after 28 days of ambient curing, which is the same as reference mix.

3.1.2 Alternative alkali sources

The one-part alkali-activation with OBA has been reported by several research groups. De Moraes Pinheiro et al. [26] investigated OBA as alkaline activator for BFS mortars. The first set of mortar samples were made with replacement of BFS by OBA from 15 to 35 wt% BFS. The second set was made with the addition of OBA from 5% to 25% wt BFS. Samples were cured in a thermal bath at 65 ºC, for 7 days. The highest 7-day compressive strength was obtained for samples with addition of 25% of OBA (38.38 MPa).

Similar results were obtained in another study by this research group [25]. Mortar samples were cured at 65ºC and 100% relative humidity, until the day of testing. The results showed that OBA activated mix had higher 3 and 7-day compressive strength than KOH activated mix (OBA: 29.9 MPa at 7 days and 45% strength gain with respect to 3-day strength; KOH: 16.9 MPa, 33% of strength gain).

The feasibility of OBA fly ash (OBFA) and OBA bottom ash (OBBA) application as activators for BFS was also evaluated by Alonso et al. [26]. Paste samples made with 70 wt % BFS and 30 wt % of OBFA or OBBA cured at 45 °C or 85 °C, had 28-day compressive strength of 33 MPa to 18 MPa. OBFA activator induced development of higher strength than

the OBBA activator. In pastes prepared with 30 % wt. OBFA, strength values were comparable to those developed by BFS pastes activated with a commercial KOH.

To activate BFS and produce mortars made only from waste materials, Font et al. [15] used OBA as alkali and RHA as a silica source. The biomass ashes were dissolved in water 24 hours prior to mixing. Authors investigated the influence of curing regime and use of OBA as addition (15, 20 and 25%) or replacement (20%) by mass of BFS. The highest 7-day compressive strength was attained for 25% addition of OBA and curing at 65 ºC, which accelerated the early strength development, resulting in up to 58.1 MPa. for the addition of 25% of OBA. Curing at elevated temperature. Mixes with both replacement and addition of 20% of OBA 7-day compressive strength was 52.1 MPa for elevated temperature curing and 35.0 MPa for ambient temperature and 100% RH curing. These mixes, cured at ambient temperature, were also tested for 3, 28, 60 and 90 days, reaching 7.8, 46.2, 61.5, 67.4 MPa, respectively.

Almond shell biomass ash has also been studied as replacement and addition to BFS (from 15% to 35% and 5% to 25% in respect to BFS mass, respectively) [34]. Mortar samples were cured for 7 days at 65ºC, and the obtained compressive strength values were in the range 25-45 MPa. These compressive strengths were higher than the once obtained by activation with 4M and 8M KOH solution.

The same research group investigated the potential use of nutshell ashes, mango seed-bark ashes and hazelnut ashes as an alkaline activator for BFS [27]. Alkali-activated pastes exhibited 26 MPa compressive strength after 28 days. The first 7 days, samples were cure at 65 ºC, after which the samples were kept at 20 ºC until the testing.

Hazelnut was also proven to be efficient BFS activator in the research of Omur et al. [28]. The experimental testing of pastes cured at 22 ºC and 50% RH showed that the compressive strength can reach up to 26.8 MPa at 28 days, which was equivalent to reference mixtures activated with 2.0 M NaOH and 2.2 M KOH.

SSA was used as an alternative activator for BFS to form grout slurries, reaching approximately 24 MPa [29]. BFA and black RHA based grouts were successfully activated with SSA and SHA reaching 24.31 MPa, 22 MPa, respectively, after 28 days of curing at ambient temperature and 95% RH (both exceeding reference KOH activated grout) [32]. In this study, WSA was also tested as alternative alkali activator, but did not exhibit good performance (9 MPa).

The authors of this paper published research results on mortars synthesized from BFS and locally available SHA, generated by the edible oil production company in the Autonomous Province of Vojvodina, Republic of Serbia. The samples reached 29 MPa after 28 days of curing [30]. The influence of curing at elevated temperature for 5 days, followed by ambient curing, was tested on the same mixes [31], where it improved early age compressive strength. However, it was proven that 28-day compressive strength of up to 30 MPa can also be reached by curing only at ambient conditions.

Peys et al. [35] investigated the possibility of using maize cob and maize stalk ashes to activate metakaolin. The pastes were prepared by two-part and one-part activation. However, the metakaolin binder produced with ash solutions as activators exhibited instability in water and low strengths. The application of maize ashes as an activator in powder form for producing one-part pastes was successful. Tested compressive strengths at 7 days were in the interval between 27 MPa and 40 MPa, depending on the ash-to-metakaolin ratio (0.9-

1.2) and curing regime (open: 48h at 60ºC and 80ºC; wrapped: 48h at 80ºC; pre cured 24h at 20ºC and then opened for 48h at 80ºC).

FA (pulverized fuel fly ash) and wood ash with high calcium content (61% CaO) and moderate potassium content (12% K_2O) were used, in different ratios, to synthesize alkali-activated pastes [36]. The compressive strength after 7 days was 12 MPa and 16 MPa after 28 days. The significantly lower strength compared to other referenced research is due to the lower potassium content, as suggested by authors. Considering the high CaO content, wood ash would possibly be more effective as a replacement for precursor.

3.2 Biomass ashes as precursors

The possibility of using wheat straw ash (WSA), RHA, SCBA, SCSA, CCA and palm oil fuel ash (POFA) as precursor due to their higher silicon content was also tested. Mixes were activated using conventional sodium hydroxide and silicate activators. Replacement of BFS with SCBA by replacing 15% and 25% resulted in a minor increase in 7-day compressive strength compared to samples with 100% BFS and decrease in 28-day compressive strength. Replacement of BFS with maximum 25% of SCSA resulted in higher compressive strengths than reference mixes. BFS and FA replacement with up to 35% of RHA also increased compressive strength, as well as 30% BFS replacement with POFA. Mixes with higher levels of replacement resulted in strength decrease [41].

The application of biomass ashes as precursors has been investigated as partial or complete replacement of metakaolin (25-100%) for production of geopolymer pastes [42]. The ashes originated from the combustion of mixed olive pruning, forest residues and energy crops. The study showed that the 50% replacement of metakaolin increased the compressive strength of the geopolymer. It was also concluded that the biomass ash contributes to the binder as an activator due to the present of soluble alkaline salts.

Alkali-activated concrete samples with 25% metakaolin, 25% FA and 50% WSA as precursor outperformed Portland cement-based system in terms of 7-days compressive strength after ambient curing (51.3 MPa vs. 32.4 MPa) [39].

4. CONCLUSIONS

The presented scoping review showed that various types of biomass ashes can successfully substitute conventional alkaline hydroxide and silicate activators in the synthesis of AAMs. Some of them are suitable as partial substitution for the precursors. Based on the inclusion/exclusion criteria, the paper presents a structured overview based on the type of precursor, biomass ash as alternative activator, compressive strength performance, and curing conditions.

The most effective alternative alkali activators are ashes rich in potassium and amorphous silica, particularly when applied to BFS- or metakaolin-based systems. Early-stage thermal curing significantly enhances strength development, although certain formulations show potential even under ambient curing.

The reviewed papers are predominantly concerning RHA-activated BFS and OBA-activated BFS systems. Only a few papers considered ternary systems, with BFS, RHA and SSA and BFS, OBA and RHA. Silicon-rich biomass ashes are usually dissolved in alkali hydroxide solutions, i.e., two-part alkali-activation process is applied. Alternative alkali

sources were mostly added in powder form (one-part alkali-activation). Samples activated with silica derived from biomass show comparable performance to those using conventional or industrial silica sources, although lower strengths can also occur. In contrast, biomass used as an alkaline source consistently outperforms chemical hydroxides.

The highest replacement of conventional precursors was achieved for RHA replacement of BFS and WSA replacement of metakaolin-FA blend. Generally better results were attained for biomass ashes used as alternative activator than precursor.

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