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Review Article

An overview of optimum calcination parameters for improved pozzolanic activity in biomass ash

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ABSTRACT

In today's construction practices, incorporation of carbon positive coal fly ash in cement manufacturing has lowered the pace of primary raw material consumption. Coal reserves are limited and hence commercially available coal fly ash will vanish in the coming future. Biomass ash is a sustainable alternative. This ash is carbon neutral and comply with the provisions of supplementary cementing material provided in IS 3812 (Part-I): 2013 and ASTM C618. Biomass ash contains considerable amount of unburned carbon. Most researchers adopt temperature controlled calcination for carbon removal but controlling only temperature does yield ash with optimum pozzolanic activity. Therefore, the present review aims to study the effects of underexplored parameters like retention time, heating rate, cooling method and combustion technology along with temperature for determining the optimum control over each parameter with the help of chemical mechanism. The study of mechanical performance of concrete, incorporating ash treated at various calcination parameter with distinct control reveals the optimum temperature, retention time, heating rate and cooling regime were 500–700°C, 2 – 5 hours, less than 10 °C/ min and rapid cooling respectively. The characteristics of ash obtained after calcination with all parameters of thermal treatment, regulated at optimum extent will possess high pozzolanic activity. The treatment might increase the cement replacement percentage and the mechanical performance of cement based matrix. The utilization of biomass ash will decelerate the pace of raw material consumption required for cement production, reduce land overburden and suffice needs of construction industry leading to a more sustainable future.

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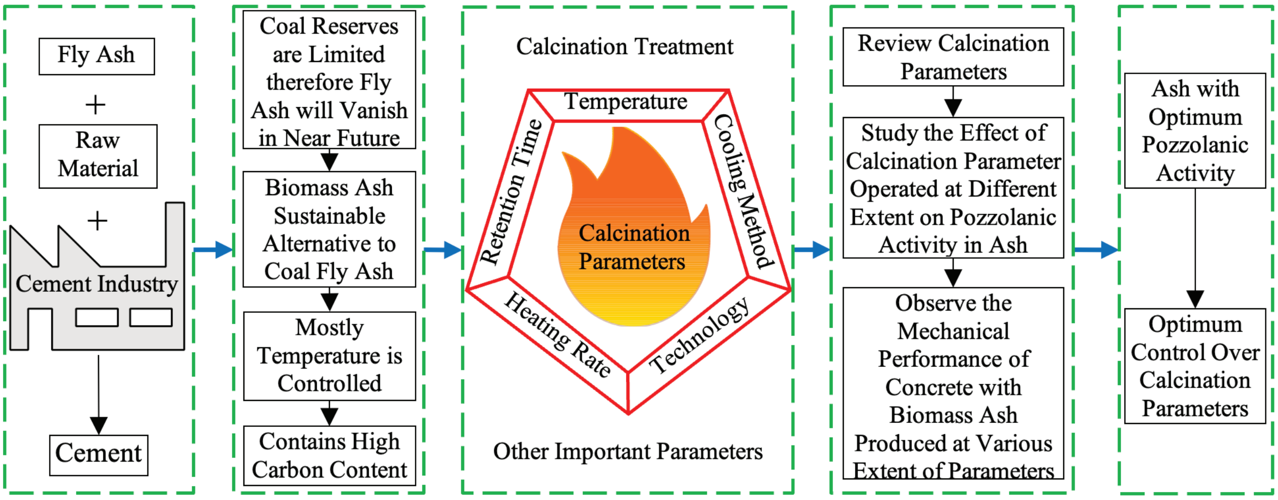
GRAPHICAL ABSTRACT



INTRODUCTION

Cement production is resource, energy and emission intensive process. To meet cement production demands natural resources are exploited but the introduction of coal fly ash in cement manufacturing has decelerated the rate of raw material consumption. Central Electricity Authority (CEA) reports 92.41% utilization of coal fly ash in the year 2021-2022 and therefore, fly ash is deemed as resource rather than a waste [1]. With fly ash's wide applicability and higher consumption rate, the non-renewable fly ash will soon vanish with the extinction of coal reserves. To cater this issue, biomass ash (BMA) is a good and promising alternative to coal fly ash. The ash generated after the combustion of biomass fuel is termed as biomass ash. Flow chart in Figure 1 depicts the production and utilization of biomass ash.

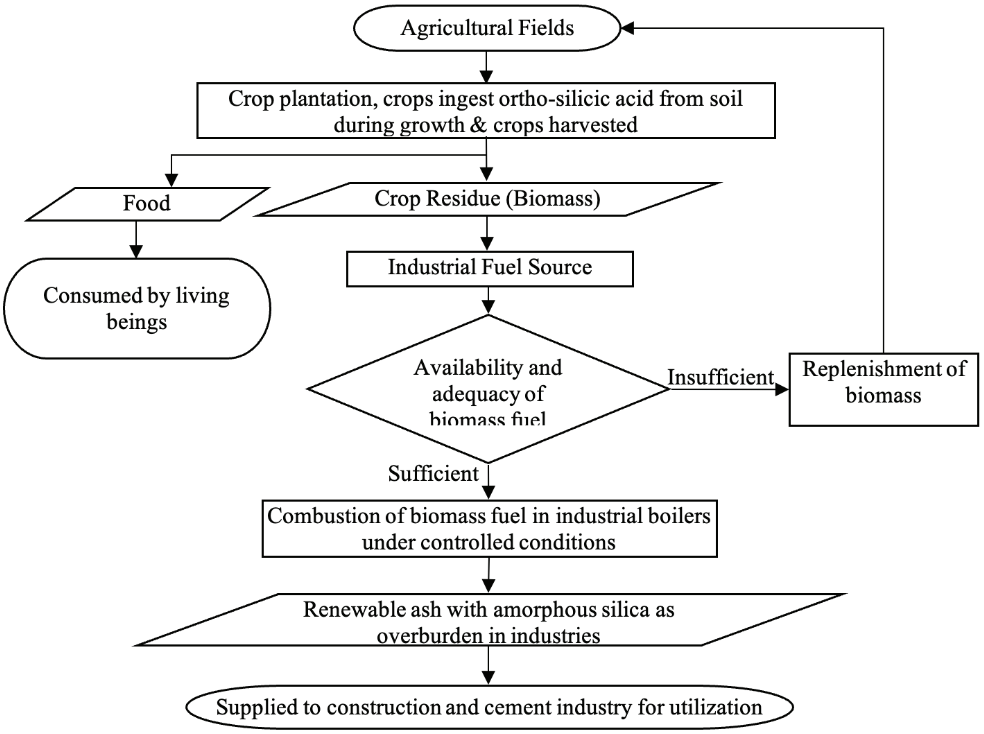


Figure 1. Flow chart showing production and utilization of renewable ash from biomass fuel.

Biomass is a promising alternative source of fuel for energy production. It is considered as carbon neutral energy source since the CO_2 emitted during the combustion of biomass is equivalent to the CO_2 absorbed during the plant growth [2-4]. A fully biomass based energy generation can reduce CO_2 up to 96%, provided substantial quantities are available throughout the year [5]. Utilization of biomass as a fuel source somewhat relieves burden of CO_2 emissions but increases ash disposal concern. The extent of ash produced globally by various biomass fuels [6-24] are shown in Table 1. The problem of ash disposal could be very well addressed by the construction industry [25]. This BMA contains reactive silicates [26], aluminates [27], alumino-silicates etc. which undergoes reaction with portlandite to form supplementary calcium silicate hydrate (C-S-H) [28] or calcium alumino-silicate hydrate (C-A-S-H) or both. These supplementary products formed reinforces interfacial transition zone enhancing the physico-mechanical behavior of concrete. Table 2 compares various properties of fly ash [29-36] and BMA [37-41].

In process of growth, crops like sugarcane [42], rice [43], wheat [42] etc. imbibe orthosilicic acid from soil and store it in plant cellular structure in amorphous form [44]. These discarded crop residues are utilized as energy source in industries where they undergo controlled calcination for heat generation or burnt in open fire (uncontrolled calcination) on agricultural fields. The ash resulting from controlled and uncontrolled calcination possess distinct characteristics. Uncontrolled calcination of biomass yields ash with greater carbon content, loss on ignition and crystalline phases[45]. This is due to inefficient oxygen supply which cause uneven local temperature along with incomplete combustion and results in inferior pozzolanic characteristics [46]. Therefore, the optimum pozzolanic behavior of ash is governed by the precise control over every calcination parameter. Any deviation in the parameters above or below the optimal control, either increases the crystallinity [47] or carbon content inside the ash. Table 3 demonstrate the effect of parameters [48-51] of calcination on quality of ashes [45,52-54].

Therefore, article intended to review the influential parameters of calcination that results in ash with high pozzolanic activity. It is already known that, thermal treatment temperature affects the properties of BMA, but there are some other parameters which are equally important for improving the pozzolanic activity in BMA. The combined effect of underexplored parameters with their optimal control during the process of thermal treatment along with temperature of thermal treatment needs to be reviewed for enhancing the pozzolanic efficiency of BMA. For finding the optimum combined effect of all these thermal treatment parameters, various properties of BMA and hardened properties of cement based matrix incorporating BMA were studied in the present review article.

Process of Biomass Combustion

Before understanding the importance of calcination parameters, it is necessary to review the calcination process which helps in determining the impact of parameters on the generated ash. Almost all biomass contains hemicellulose, cellulose and lignin in variable proportions. These organic compounds are composed of carbon, oxygen and hydrogen [55]. When biomass is subjected to calcination, thermal decomposition takes place in the following steps, a) heating b) drying c) de-volatilization to produce gases and tar d) combustion of volatile matter e) combustion of charcoal (char) [56]. When this biomass is subjected to a cycle of elevating temperature (calcination), these organic contents at first undergoes heating and subsequent drying (130-150°C) and starts de-volatilization producing gases oxygen, carbon-di-oxide, methane and carbon-mono-oxide (O_2, CO_2, CH_4 and CO) [57] and tar in a temperature range of 130-380°C [58]. Tar is composition of more than 100 compounds [59]. The combustion of volatile matter and carbon occurs simultaneously but removal of carbon occurs at higher temperature and consumes a longer duration. The volume of volatile matter in biomass fuel (BMF)

Table 1. Global annual production of biomass and ash from distinct sources

Sr. No.	Crop	Residue as fuel source	Crop/product production (million ton)	Biomass production (million ton)	Ash production (million ton)	References
1	Sugarcane	Bagasse	1900	494	12.6	[6,7]
2	Rice	Husk	518	145.04	29	[8-11]
		Straw		777	116.55	
3	Wheat	Husk	772	154.4	21.5	[12-15]
		Straw		1080.8	72.4	
4	Maize	Corn cob	1070	203.3	4.67	[16-18]
5	Palm Tree	Shell, fibre, fruit bunch	72.27	650.43	26	[19,20]
6	Coconut	Shell	62.46	9.369	0.17	[21,22]
7	Groundnut	Shell	43.98	17.59	0.44	[23,24]

Table 2. Comparison between coal fly ash and BMA

Description	Biomass Ash	Coal Fly Ash
Source	The major sources of biomass are agricultural residue, municipal solid waste, wood and dried cattle waste [37]. The ash resulting from biomass might be high as 43.3% for a particular source but on an average, it is 6.00% [38].	There are four different ranks of coal which produces fly ash after combustion. They are Anthracite, Bituminous, Subbituminous and Lignite [29]. The ash resulting from high impurity coal may be as high as 48.4% but on an average, it is 19.8% [30].
Chemical Characteristics	The chemical composition of biomass ash depends on the type of biomass. However, ash from woods and agricultural waste contain silica (SiO_2: upto 94.48%) and calcium oxide (CaO: upto 83.16%) in high quantities and alumina (Al_2O_3: upto 15.12%), and iron oxide (Fe_2O_3: upto 9.54%) in less quantities. The alkali oxides which are considered as impurities in cement-based products are present in very high proportions in some of the BMA. It is observed that, Na_2O and K_2O in BMA could be as high as 29.82% and 53.38% [37].	Two different fly ash evolve from the combustion of coal, i.e. class C and Class F. Class C fly ash contain CaO greater than 10%, whereas Class F fly ash possesses CaO less than 10%. The other major mineral oxide which is found inside Class C & F fly ash are silica (SiO_2: 20-80%) and alumina (Al_2O_3: 1-55%) and iron oxide (Fe_2O_3: 1-44.7%). The ash also contains alkali (Na_2O & K_2O) in quantities varying from 0.0 to 7.3% [31]. The loss on ignition for coal fly ash ranges between 0.0 – 32.8% [32].
Physical Characteristics	The fineness of BMA can be as high as 700 m^2/kg [39] and specific gravity ranges between 1.82 – 2.46 [37]. The particle size and loos bulk density of BMA varies between 0 – 10 mm [37] and 430 – 960 kg/m^3 [40]. The particles of almost all the BMA are irregular in shape and their particles possess inherent porosity [26,41].	The fineness and specific gravity of coal fly ash vary between 231.5 – 523.9 m^2/kg and 1.94 – 2.72 [32]. The particle size and loose bulk density of coal ranges between 1 – 100 µm and 540 – 860 kg/m^3 [33]. Coal fly ash particles are spherical in shape and exhibit a porous/non-porous nature [34,35].
Environmental Impact	Biomass is a carbon neutral fuel source. BMA resulting from the power plants and other industries is landfilled. The ash generally does not possess very hazardous elements and therefore can be used in fertilizers etc [38].	Coal is a fossil fuel and therefore it has a high carbon footprint which contributes significantly to the GSG emission. The coal fly ash is generally landfilled and its leaching causes hazardous elements like Arsenic, Boron, Chromium, Cadmium, Lead and Mercury pollutes the surrounding soil [36].

Table 3. The effect of parameters of calcination on quality of ashes

	Type of Calcination	Uncontrolled	Controlled		
Features of Biomass Calcination	Control Over Calcination Parameters	No Control	Control Below Optimum	Optimum Control	Control Above Optimum
Properties in Ash	Carbon Content in Ash	High [48]	High [49]	Very low [50]	Very low [51]
	Nature and Reactivity in Ash	Crystalline and Less Reactive [45]	Amorphous and Less Reactive [52]	Amorphous and Highly Reactive [53]	Crystalline and Less Reactive [54]

can be as high as 85 % [60] and combustion consumes around 40-50 % of the total burnout time which occurs at higher combustion rate than char. Whereas combustion of char depends on temperature, residence time, contaminants producing inhibiting and catalytic effect, oxygen concentration and surface area of fuel particle [61].

Generally, the thermal decomposition of hemicellulose (150-350°C) and cellulose (275-350°C) occurs at a lower temperature but decomposition of lignin occurs in wide temperature range (275-500°C) [62]. Thus, these organic compounds experience thermal disintegration at a temperature below 500°C and decomposes into tar and char (forms of carbon). Further increase in temperature, the carbon undergoes oxidation at a faster rate, rather at temperature below 500°C where volatilization is faster [63]. For decomposition of char, oxygen present in the environment is transferred onto the particle surface where it diffuses into the porosity of char. The chemisorption of oxygen on the active sites causes decomposition and releases CO. CO chemically combines with O_2 from the gaseous boundary layer and releases CO_2. The oxidation of carbon occurs continuously as more and more reactive sites are exposed [64]. Also, it was reported that the molecules of H_2O and CO_2 dissociates into their free radicals which are absorbed by the active sites and tends to oxidize carbon [65]. However, following chemical reactions are proposed by [66] for oxidation of carbon particles.

$$\mathrm{C} + \frac{1}{2}\mathrm{O_2} \rightarrow \mathrm{CO} \tag{1}$$

$$\mathrm{CO} + \tfrac{1}{2}\mathrm{O_2} \rightarrow \mathrm{CO_2} \tag{2}$$

$$\mathrm{CO_2} + \mathrm{C} \rightarrow 2\mathrm{CO} \tag{3}$$

$$\mathrm{C} + \mathrm{O_2} \rightarrow \mathrm{CO_2} \tag{4}$$

$$\mathrm{H_2O} + \mathrm{C} \rightarrow \mathrm{CO} + \mathrm{H_2} \tag{5}$$

$$\mathrm{CO} + \mathrm{H_2O} \rightarrow \mathrm{CO_2} + \mathrm{H_2} \tag{6}$$

Important Parameters for Controlled Calcination

Temperature, retention time, heating rate, cooling method and combustion technology are the parameters that affects carbon removal efficiency, crystal structure and colour of ash in calcination. These parameters cannot be regulated in uncontrolled calcination. A control over these parameters modulates the decomposition of volatile matter and char in biomass [67]. Therefore, a discussion about these parameters is elucidated in succeeding sections.

Calcination temperature

Calcination temperature is the primary factor that induce good pozzolanic activity in ash for utilizing as a supplementary cementitious material (SCM) in concrete. Results on BMA characteristics are available from temperature range of 300°C to 1000°C. A temperature over 500°C mostly yields amorphous ash with low carbon content whereas temperature below 500°C results in amorphous ash but with high carbon percentage [60]. The two main phases that govern carbon decomposition are (a) oxygen diffusion rate (b) chemical reaction rate. Oxygen diffusion rate is responsible for amount of carbon decomposition whereas chemical reaction rate determines the pace of carbon decomposition. Combustion at lower temperatures (400 - 750°C), oxygen diffusion rate is greater than chemical reaction rate. In the mid order temperature range (750 - 900°C), the oxygen diffusion and chemical reaction rate are almost similar whereas in high temperature range (900 - 1100°C), the chemical reaction rate overrides oxygen diffusion rate [68,69].

Most researchers have utilized low to moderate temperature zone for effective biomass combustion. These two zones have constantly been reported to produce amorphous silica. Crystallization of silica mainly occurs when temperatures exceed beyond 700°C. The impurities present in biomass interact with amorphous silica at high temperatures and transforms it into crystalline phases [70]. According to Ibrahim and Helmy, the phase transformation of ash begins at 725°C and at a temperature around 800°C the ash is readily transformed into Crystobalite. Calcination at lower temperature results in amorphous (disordered) Cryatobalite in ash. The temperature increase causes nucleation of silica, which occurs simultaneously with crystallite growth. Nucleation of silica particle was predominant in temperature range of 800-900°C while crystallite growth and perfection was favoured within 1000-1100°C [49]. In another experimentation conducted by Hanafi et.al. on rice husk ash for the study of surface characteristic stated an increase in surface area and total pore volume due to destruction of silanol (destruction of bond between Si and OH) group in a temperature range of 500-600°C. But a further increase of temperature beyond 600°C upto 1000°C reduced the surface area, total pore volume and increased crystallite size. A drastic reduction in surface area, total pore volume and increase in particle size was more pronounced at 1000°C while till 900°C temperature ash also contained amorphous phases which was evident from the X-Ray diffraction (XRD) pattern. At 1000°C temperature, disordered Crystobalite had transformed into crystalline state which was marked by decrease in surface area, total pore volume and increased particle size. These results were found consistent with Ibrahim and Helmy. Further increase of temperature to 1100°C increased surface area and total pore volume, and declined particle size due to formation of disordered Tridymite phases. The escalation of temperature further till 1400°C declined the surface area and total

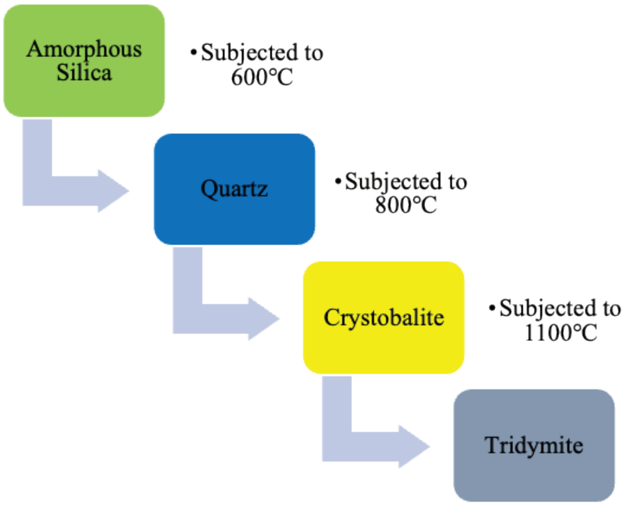


Figure 2. Phase transformation of silica in biomass.

pore volume [71]. Similar results were obtained by Hamad and Khattab where they confirmed that crystallization of Crystobalite occurred at 800°C while phase transformation of Crystobalite to Tridymite initiated at 1150 °C. Amorphous Crystobalite phases were present below 600°C. [72]. The structural transformation of silica extrapolated from review is shown in Figure 2. Therefore, treatment of biomass in low temperature region will possibly retain the amorphous nature of ash.

However the transformation of silica varies from one biomass to other biomass type. This is generally due to the varying chemical composition of different BMA. In rice husk ash (RHA) silica (SiO_2) is present in major quantities whereas other oxides and impurities are present in minor amount. The impurities play a pivital role in the phase transformation of silica at higher temperature [49]. In another type of BMA like wood ash, where silica is present in lower amounts and calcium oxide in higher amount, the possibility of silica phase transformations is lower. The calcium oxide along with other mineral oxide have a tendency to fuse with silica and form a multiple element oxide compound [73].

Retention time

The second most critical factor concerning the calcination of biomass is retention time. When biomass is subjected to a specified temperature control it has to be retained inside the calcination chamber for the completion of undergoing chemical processes. The chemical process includes reduction of organic matter (into gases, tar and char) and oxidation of carbonaceous matter. The state of completeness of these reactions can be identified from the ashes. These chemical reactions for its completion require a specified amount of time which is known as retention / residence / holding time. When the biomass is exposed to shorter retention time these reactions are incomplete rendering unwanted characteristic in ashes but when sustained at optimum retention time yields ash with best pozzolanic activity. When the temperature is maintained between 400-600°C for a residence time of 3-6 hours it yields ash with low carbon content and crystallinity [74,75]. The primary necessity of retention time is removal of carbon. From the studies conducted so far on the calcination of biomass it can be stated that the temperature and retention time are inversely related to each other. Siddique makes an affirmation to this inverse relationship stating that amorphous silica ash could be produced with a prolong retention time if the temperature is maintained below 500°C. If the temperature is raised to 900°C and 1000°C the ashes are still amorphous but for a residence time of less than 1 hour and 5 minutes respectively [76]. An experimental investigation was conducted by Vayghan et.al to study the effect of retention time on physic-chemical properties of rice husk ash. The experimentation was conducted at 700°C with retention time of 0.25, 1, 4, 8, 16, and 32 hours. With a longer retention time of even 32 hours the ash still remained amorphous which was evident from the X-Ray diffraction pattern [77].

Rate of heating

From the aspect of carbon removal, heating rate is a very crucial parameter. The impurities present inside the biomass do not allow easy oxidation of carbon instead forms an agglomerate with silica which leads to high percentage of carbon quantity and reduced surface area. In most of the research, the authors have used variable heating rate values ranging from 1°C/min to 10°C/min. The most commonly deployed heating rate was 10°C/min [78]. Rice husks when heated at a rapid rate do not allow the oxidation of carbon as desired mainly due to two probable reasons. Firstly, at higher heating rates the desired temperature is achieved very fast. Though the oxidation of carbon starts at 350°C but have a tendency to oxidize slowly and results into greater amount of carbon in ash [60]. Secondly, the intervention of impurities (K_2O) during calcination produces ash with greater carbon content [79]. Chandrasekhar et.al explored the effect of heating rate on the pozzolanic reactivity of rice husk ash. The heating rates were 1, 2, 3, 5, 7 and 10°C/min and the temperature was maintained at 700°C. With increase in heating rates the lime reactivity test displayed lower reactivity values due to conversion of amorphous silica into crystalline phase. Rapid heating resulted in ash with blackish appearance due to fixed carbon [80].

Cooling Method

The cooling method governs the phase transformation (amorphous to crystalline) inside the ash. After subjecting the biomass to optimum temperature and residence time might not yield BMA with complete amorphous phases as a result of applied cooling method. Generally, rapid and slow cooling techniques are followed. For rapid cooling the ash is quenched rapidly in air or water whereas for slow cooling the ash remains inside the heated furnace till it attains the room temperature (oven cooling) [81,82]. A better pozzolanic behavior is observed when ash is rapid cooled than slow cooling [83]. In an experimentation carried out by Vayghan et.al. [84], the effect of cooling method was studied after retaining the ash at 700°C temperature and residence time (15 min, 1, 2, 4, 16 hrs). It was deduced that, if the ash is rapidly cooled from its combustion temperature to room temperature, no crystalline phases are formed even after exposing ash till a retention time of 32 hours and with a high amount of K_2O. It was also stated that, prior to calcination if the rice husks are acid leached, K_2O is discarded, thus preventing crystallization of amorphous silica in slow cooling regime [84]. Study conducted by Nair et.al. demonstrated that, pozzolanic activity of rapidly cooled samples was more than slowly cooled ash. Also, the size of particle in quick cooling is lesser than slow cooling. This was indicative of the condensation reaction that took place in slow cooling regime of ash which rendered it as less reactive due to decreased particle surface area [85].

Technology of Calcination

The characteristics of BMA is governed by biomass combustion technology at the industries. Moving grate firing system (MGFS), fluidized bed combustor (FBC) and suspension burner system (SBS) [86] are the recognized technologies used for fuel combustion. Such technologies are potentially utilized in paper industries [87], biomass-based power plants, sugar industries and co-generation plants [88], distilleries, chemical industries etc. The most rudimentary and often utilized system for firing industrial boilers is MGFS. Lower installation, operational and maintenance cost attracts industrialists with MGFS [89]. On the contrary MGFS is usually designated as high carbon in ash, low efficiency and high emission technology [89]. The inefficient combustion of biomass is due to lower residence time than required for complete combustion of char in biomass. This not only results in greater extent of unburnt carbon but also gives away high emissions [90]. FBC is considered as a mature technology due to its flexibility, very high efficiency of fuel combustion and lower environmental impact [91]. This system offers a very high combustion efficiency between 97-98.4% which results in ash with low carbon content [92]. When any biomass is introduced in FBC high heat and mass transfer property of fluidized sand bed allows quick energy transformation at nearly constant temperature condition. The high reaction surface area available inside FBC results in high combustion efficiency and lower operating temperature than MGFS. Moreover, the excess air supplied tends to retain fuel particles in combustor for sufficient residence time which bring about complete oxidation of carbon. Contrary to FBC, the non-uniform air fuel ratio give rise to high unburnt carbon in ash and thus affecting combustion efficiency [93]. SBS is also said to produce good quality of ash but its sensitivity towards varying fuel composition significantly affects its performance. Every time the fuel inside should be finely pulverized [89].

Effect of Calcination Parameters on Chemical Composition of BMA

Biomass when subjected to calcination, it undergoes decomposition. A variation in these parameters alter the chemical composition. Table 4 shows the chemical composition of BMA with different parameters of calcination. The results of chemical composition are taken from the X-ray fluorescence (XRF) data available in research articles.

Table 4. XRF results of various BMA with different parameters of calcination

T °C	RT Hrs	HR °C/min	CM	Chemical contents									
				SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI	Ref.
Rice Husk Ash													
300	3	10	Slow	35	1.87	1.56	11.2	2.91	8.96	21.9	1.66	-	[52]
	9			48	1.57	1.55	9.28	2.52	8.35	19.35	1.39		
500	2	8	-	89.89	0.11	0.28	1.05	0.13	0.4	2.92	-	-	[74]
	2	10	-	91.56	0.19	0.17	1.07	0.65	0.47	3.76	0.16	-	[75]
	3	10	Slow	66.45	3.68	0.85	3.36	1.6	0.54	5.68	2.2	-	[52]
	6	5	-	92	0.25	0.14	0.81	1.05	0.96	3.44	-	3.62	[76]
	7	10	Slow	73.05	1.16	1.18	3.5	1.45	0.47	5.67	2.74	-	[52]
600	1	5	-	92.09	0.1	0.07	0.97	0.53	0.55	4.04	0.11	1.52	[77]
	2			93	0.1	0.07	0.92	0.45	-	3.62	0.46	1.48	
	2	20	-	81.4	0.26	0.93	2.42	1.02	1.47	6.79	0.18	3.13	[78]
	3	10	-	90	0.68	0.42	1.23	0.35	-	2.8	0.32	-	[79]
	6	-	Slow	88.84	0.8	0.39	1.78	0.92	0.35	2.8	1.1	2.02	[80]
650	1	3.33	-	87.32	0.22	0.28	0.48	0.28	1.02	3.14	1.02	2.10	[48]
	-	2	Slow	92	0.31	0.38	0.97	0.47	-	3.87	0.2	0.76	[50]

Table 4. XRF results of various BMA with different parameters of calcination (continued)

T °C	RT Hrs	HR °C/min	CM	Chemical contents									
				SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	LOI	Ref.
Rice Husk Ash													
700	1	3	-	89.03	2.23	0.66	0.96	0.38	-	2.34	2.59	0.7	[81]
	1	10	Rapid	88.07	1.35	0.22	1.04	0.74	0.49	2.02	1.15	2.61	[82]
	2	-	Slow	84.95	0.45	0.32	0.84	0.4	-	0.21	0.5	9.81	[83]
	3	10	Slow	88.00	0.38	0.41	2.1	0.52	1.98	4.35	0.32	-	[52]
	4	10	Rapid	94.91	0.05	0.25	0.98	0.44	-	1.67	0.07	1.28	[84]
	5	10	Slow	94.63	0.18	0.19	1.05	0.18	0.62	2.18	0.15	-	[52]
	6	5	-	91.15	0.41	0.21	0.41	0.45	0.62	6.25	0.05	0.45	[85]
	6	10	Slow	93	0.2	0.13	0.49	0.73	0.15	1.3	0.02	3.98	[54]
	8	10	Rapid	95.22	0.09	0.19	1.16	0.39	-	0.14	0.03	0.8	[84]
	16	10	Rapid	95.1	0.05	0.11	0.1	0.41	-	1.66	0.05	0.92	
800	-	-	Slow	89.5	0.4	2.86	0.3	0.25	-	-	-	4.0	[86]
			Rapid	84	1.39	2.1	0.6	0.85	-	-	-	5.85	
900	3	10	Slow	92	0.23	0.21	1.39	0.36	1.42	2.99	0.2	-	[52]
	4	5	Slow	98.19	0.19	0.02	0.18	-	-	1.04	-	0.5	[87]
	6	5	-	92.0	0.37	-	1.05	0.95	1.06	3.2	-	0.29	[76]
Sugarcane Bagasse Ash													
550	2	10	Slow	58.37	7.24	4.04	3.07	3.02	-	8.20	0.05	25.1	[88]
	3	10	Slow	64.00	7.46	3.50	2.46	3.15	-	7.71	0.04	18.5	
600	2	10	Slow	64.14	6.82	5.12	1.78	1.75	-	6.32	0.22	5.83	
	3	10	Slow	69.79	9.86	3.02	1.92	1.78	-	5.57	0.05	5.61	
	3	10	-	69.6	15.7	5.7	1.3	-	1.5	2.2	-	2.1	[89]
650	2	10	Slow	64	8.16	5.04	1.08	1.90	-	6.55	0.25	3.90	[88]
	3	10	Slow	72.5	10.63	4.04	1.14	1.34	-	3.56	0.08	3.57	
700	2	10	Slow	63.86	9.5	4.96	1.81	2.04	-	6.78	0.27	3.22	
	3	10	Slow	74.22	9.87	4.13	1.72	0.96	-	2.7	0.03	2.46	
750	2	10	Slow	66.32	12.97	5.27	1.35	1.6	-	4.7	0.36	3.12	
	3	10	Slow	75.91	10.49	2.99	0.84	1.31	-	3.3	0.07	3.13	
Wheat Straw Ash													
550	4	-	-	62.5	9.08	1.67	5.90	-	1.66	17.05	-	-	[90]
	8	-	-	60.07	6.98	1.45	8.47	-	1.67	15.87	-	-	
800	0.5	-	-	55.5	11.24	2.79	7.40	-	1.84	22.43	-	-	
Corn Cob Ash													
350	3.75	-	-	10	1	2.3	4.2	10	-	57.5	-	-	[91]
400	3.75	-	-	10.7	1.5	2.5	4.4	10.1	-	57	-	-	
450	3.75	-	-	12.4	1.7	2.7	5	9	-	56	-	-	
500	3.75	-	-	14	1.9	2.8	7	9.5	-	52.5	-	-	
550	3.75	-	-	15.1	1.9	2.9	6.9	9	-	52	-	-	
600	3.75	-	-	18.3	2.7	3.12	8	10.2	-	47.2	-	-	
	4	-	-	63.91	4.01	3.95	4.13	2.91	0.88	12.12	-	-	[92]
650	3.75	-	-	20.2	3.1	3.7	10	8.4	-	45.4	-	-	[91]
	5	-	-	59.66	4.92	-	1.86	1.68	-	15.48	0.34	-	[93]
	8	-	-	66.38	7.48	4.44	11.5	2.06	1.07	4.92	0.41	-	[94]
700	3.75	-	-	20	2.9	3.5	11	8.2	-	45.3	-	-	[91]

Where, **T** = Temperature, **RT** = Retention time, **HR** = Heating rate and **CM** = Cooling method

The variation in chemical composition could be observed in terms SiO_2, impurities and loss on ignition (LOI). The criteria of SCM defined by IS 3812-1: 2003 and ASTM C-618 states that the summation of oxides (SiO_2 + Al_2O_3 + Fe_2O_3) should be greater than 70% [95,96]. It is observed that biomass calcined at temperature below 500°C could not qualify the SCM criteria defined by the standards. Moreover, impurity content in ash was very high. But with increasing temperature, the impurities started to decline which resulted in an increase in silica and alumina content. A similar scenario of increase in silica and alumina content was observed with increase in retention time. From the table it is also observed that, not all the ashes were able to qualify the criteria of SCM mentioned in IS 3812-1: 2003 and ASTM C-618. This is due to the presence of impurities. The impurities like K_2O were present in very high quantities (57.5%), and such ashes could adversely affect the properties of cement based matrix. At temperature greater than 600 °C and retention time greater than 3 hours the loss of ignition was found within the limits prescribed by IS 3812-1: 2003 and ASTM C-618. The limits for LOI defined in IS and ASTM are 5% and 6% respectively.

PROPERTIES OF CONCRETE

Effect of Calcination Parameters

Inclusion of SCM into concrete mixes, the mechanical concrete characteristics are influenced by the quality of SCM. Uncontrolled calcination of biomass produces ash with high carbon content and crystalline silica phases. Incorporation of such ash results into uneven mechanical characteristics of concrete. This is generally due to inconsistency of combustion parameters in open fire. BMA evolving from controlled combustion leads to predictable mechanical concrete behavior. A study of calcination factors affecting the quality of BMA and resulting mechanical behavior of concrete, guides in optimizing calcination parameters.

The foremost parameter that overrides is calcination temperature. From section 3.1 it is already clear that minimum temperature for desired ash characteristic should be greater than 500°C. The effect of inclusion of RHA treated at different temperature on mechanical properties of concrete could be seen from Figure 3. The increase in percentage compressive strength from the study of different researchers was found to be highest within the temperature region of 500-700°C which could be clearly observed from Figure 3(a). Its highest percentage increase in compressive strength was 43.05% at 700°C [84]. Considering peak at each temperature the trend shows an increase in strength till 700°C and a sudden downfall thereafter. These authors have either experimented with a particular temperature or over a range of temperature. The peak increase in percentage compressive strength reported from their investigation are 10, 21, 20.15, 3.13 and 12% at 300, 500, 600, 800 and 900°C respectively. When temperature reached 1000°C the increase in compressive strength was -0.4% which indicated that strength was below control concrete strength [52,74,97,98]. These authors reported similar trend for percentage increase in flexural and split tensile strength which could be noticed from Figure 3(b) and 3(c) respectively.

A review conducted from research of various authors revealed 700°C as the optimized temperature, therefore it was decided to observe the effect of retention time and heating rate at 700°C from available literature. After a compilation of results from different research, it could be deduced that a retention time of 2-5 hours is sufficient to obtain good pozzolanicity in ash which probably results in percentage increase in strength greater than 20%. This could be observed from Figure 4. At 500°C, Faried et.al reported 18.8% and 19.4% increase in compressive strength for a retention time of 3 and 7 hours [52]. Huang et.al. showed the compressive strength elevated by 9.67% for a retention time of 2 hours [75]. For a temperature of 600°C and 800°C, Xu et.al and Khan et.al. recorded increase in compressive strength by 12.5% and 3.13% for the respective retention duration of 2 and 5 hours [78,97].

At the considered optimized temperature (700°C) Figure 5 manifests percentage increase in compressive strength from the experimentation of different researchers at various duration of retention for heating rate of 10 and 20°C/min. At temperature of 500°C and a heating rate of 1.67, 8 and 10°C/min Khalaf and Yousif, Muthukrishnan et.al and Huang et.al displayed a percentage increase in compressive strength as 19.6, 21 and 9.67% respectively [74,75,100]. Khan et.al and Xu et.al conducted investigation at 800 and 600°C where they found the percentage increase in compressive strength as 3.13 and 12.85% with a respective heating rate of 3.33 and 20°C/min [78,97]. Khan et.al calcined rice husk at 800°C where rapid cooling was carried out at temperature of 21±1°C and for slow cooling the sample was retained in furnace. The pozzolanicity test conducted revealed a superior reactivity of rapidly cooled ash with 28 days compressive strength as 21.10MPa [86].

Effect of Varying BMA Content

Vayghan et.al. obtained optimally calcined dry acid leached RHA at 700 °C temperature an 8 hours of retention time. 0, 5, 10 and 15% RHA were the different proportions of RHA used to replace cement in the mortar matrix. The results showed that mortar with 10% RHA had the highest compressive strength [84]. Huang et.al. substituted RHA with cement at 0, 5, 10, 15, 20, 25 and 30%. The ash was treated at 500 °C temperature, 2 hours of retention time with 10 °C/ min heating rate for developing ultra-high performance concrete (UHPC). The compressive strength of UHPC at 28 days achieved was 136.6 MPa with 25% RHA [75]. Faried et.al. calcined rice husk at four different temperatures and retention time with a heating rate of 10 °C/ min. Temperature of 700 °C with 5 hours of retention time were the optimum calcination parameter. RHA replaced

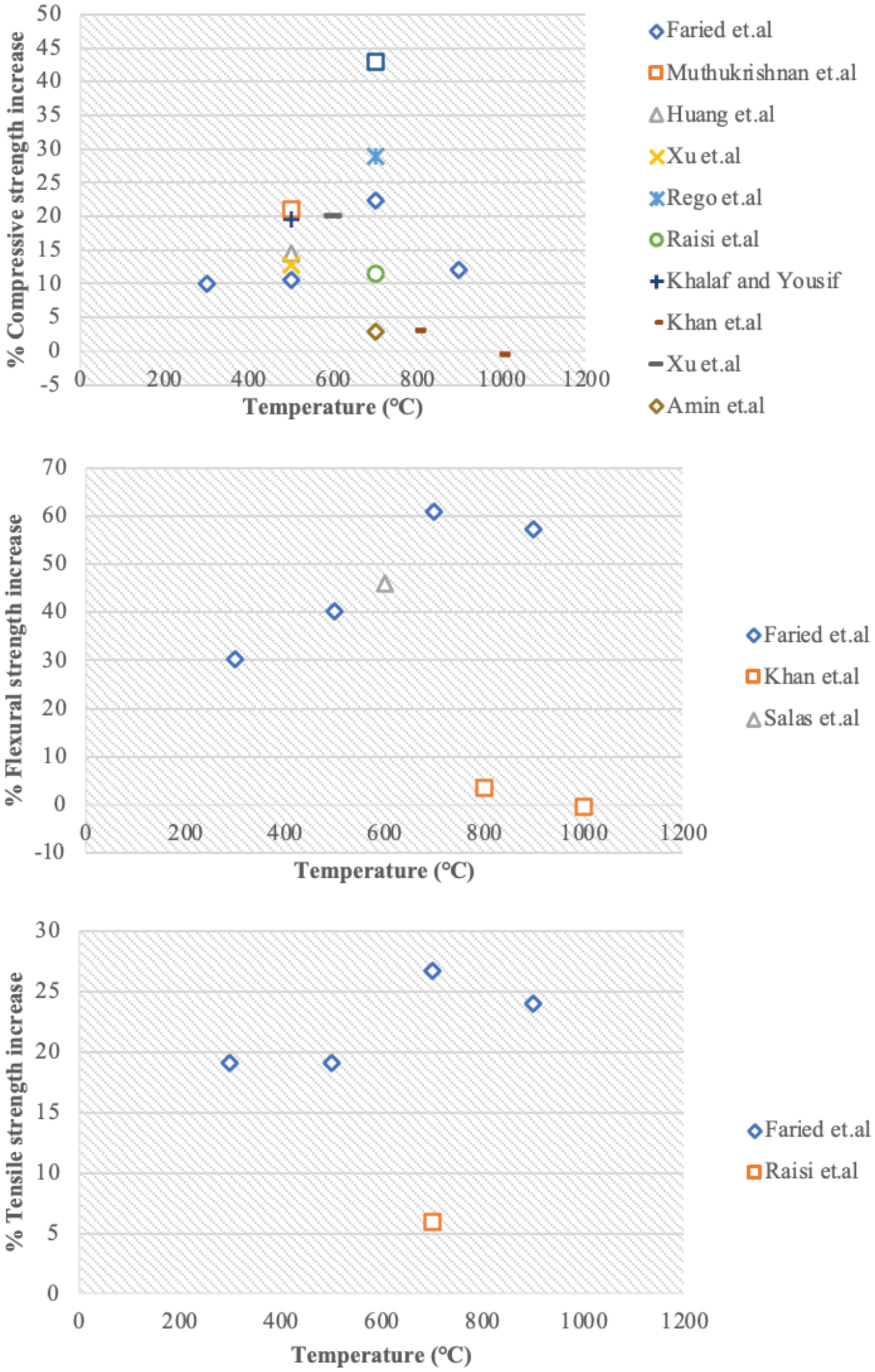


Figure 3. RHA treated at various temperature and its effect on mechanical properties (a) percentage increase in compressive strength (b) percentage increase in flexural strength (c) percentage increase in split tensile strength [52,74,75,78,82,83,97-100]. [created by author].

cement by 0, 1, 3 and 5% for making UHPC. The highest strength achieved was above 140 MPa at 3% cement replacement [52]. Amin et al. used thermally treated RHA at 700 °C it for developing concrete in three different cement replacement percentage (10, 15 and 20%). The maximum strength at 28 days was achieved for 10% concrete mix [99]. Raisi et. al. obtained RHA at 700 °C temperature, 1 hour retention time and 10 °C/ min heating rate. Five different proportions of RHA (0, 5, 10, 15 and 20%) were used to replace cement in self-compacting concrete (SCC) mixes. The highest compressive strength achieved was 57.7 MPa at 5% cement replacement [82]. As per Khalaf and Yousif

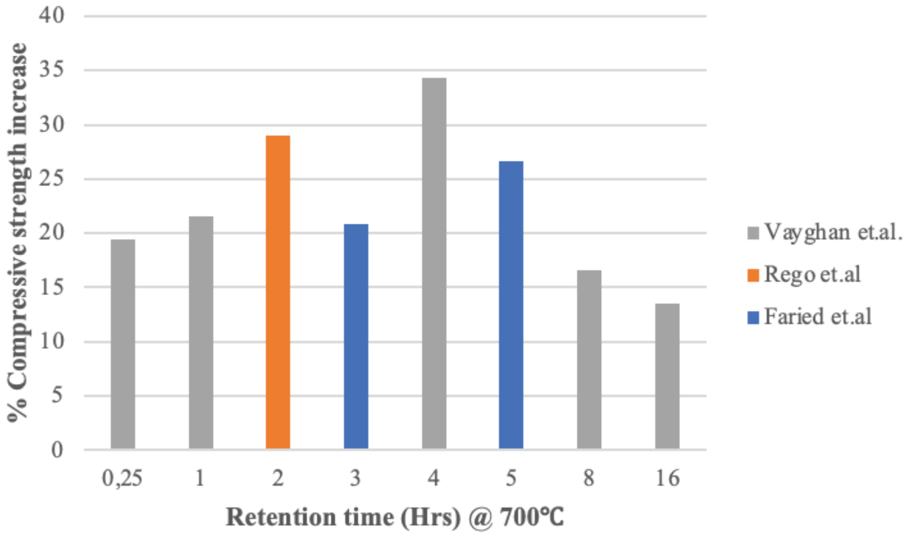


Figure 4. RHA treated at various retention time and its effect on percentage compressive strength at considered optimized temperature [52,83,84]. [created by author]

the optimum temperature and retention time of thermal treatment was 500 °C and 2 hours. The ash prepared with these optimum treatment parameters was used in mortar replacing cement by 0, 10, 20, 30, 40 and 50%. Mortar with 10% cement replacement yielded the highest compressive strength [100].

Synergistic Effect of Biomass Ash with Different Fibers

Hamzeh et.al. studied the combined effect of RHA treated thermally at 700 °C with three different derived fibers. The fibers were derived from virgin kraft pulp (VKP), fiberboard (FFB) and old corrugated containers (OCC). The cement paste cubes were used to observe the effect of RHA and fibers. The proportion of fibers to cement was constant (25: 75) and different proportions of RHA were 0, 25 and 50% by weight of fibers. The cubes attained highest compressive strength with OCC fibers with 25% RHA replacement [101]. Hesami et.al. burned rice husks in an open fire for 2 hours and then and used a special furnace to decompose the carbon in RHA. Seven different percentage of RHA (0, 2, 4, 6, 8, 10 and 12% by cement weight) along with three distinct fiber types were used to develop pervious concrete for pavement. Glass, steel and polyphenylene (PP) fibers with a corresponding volume percentage of 0.2, 0.5 and 0.3% to the volume of concrete were used in the investigation. It was observed that the performance of

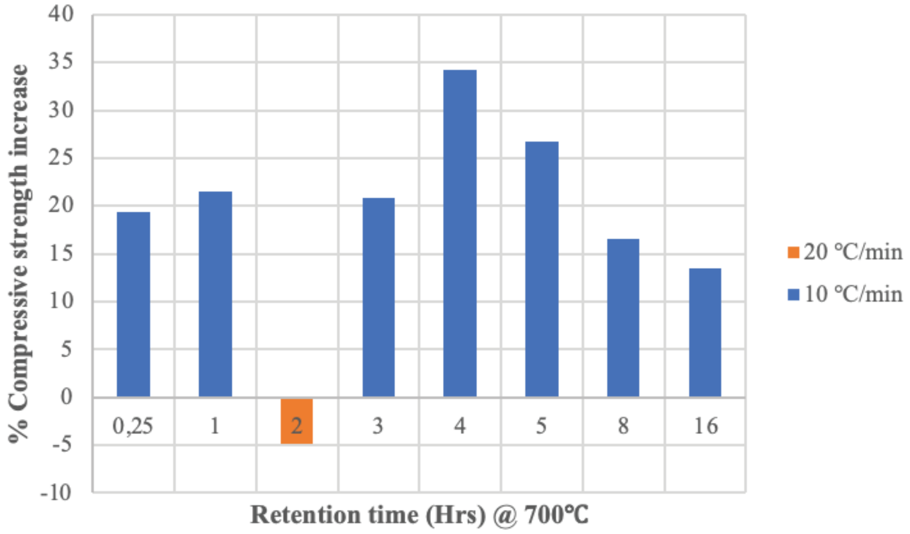


Figure 5. RHA treated at various heating rate and its effect on percentage compressive strength at considered optimized temperature and over a range of retention time [52,84,98]. [created by author]

fiber concrete with RHA was greater than control. The best mechanical performance of pervious concrete was shown by PP fibers at RHA percentage of 8% [102]. Memon et.al. investigated the significance of thermally treated RHA along with polypropylene (PPY) fiber on various properties of concrete. The procured RHA was thermally treated at 700°C for one hour in an electric furnace and substituted cement by weight percentage of 0, 5, 10, 15 and 20%. PPY fibers were added to concrete in four different percentages (0, 0.2, 0.25, and 0.3). The mechanical performance of concrete with 10% RHA and 0.25% PPY fibers was superior than control and other mixes [103].

Koushkbaghi et.al. developed steel fiber reinforced concrete with RHA as SCM and recycled coarse aggregates (RCA). Rice husk pellets were calcined in an electric furnace between 500 - 700°C, for 6 hours with a heating rate of 5 °C/ min for obtaining RHA. The 20% RHA and 1.5% steel fibers were added to the concrete. RCA replaced natural coarse aggregate by 0, 50% and 100%. The increasing percentage of RCA lowered the mechanical performance of concrete, whereas adding 20% RHA and 1.5% steel fibers increased the mechanical performance of concrete when compared with control concrete. The mechanical performance of concrete with 20% RHA (no steel fibers) was greater than 1.5% steel fibers (no RHA) [104]. Sobuz et.al. developed self-compacting concrete (SCC) with RHA and fibers derived from waste galvanized copper wire. SCC included 2% RHA and 0, 0.25, 0.5 and 1.0% fibers. The results manifested that the mechanical performance of control concrete was better than SCC with inclusion of RHA and fibers. The compressive strength of SCC with 2% RHA and 0.25, 0.5 and 1.0% fibers was 17.99, 18.09 and 19.7 MPa whereas for control (0% RHA and 0 % fibers) SCC it was 22.62 MPa [105]. Silva and Naveen carried out investigation on combined effect of RHA and coconut coir (CC) fibers in cement mortar. The RHA from brick kiln was used to substitute cement from 0 – 20% with a replacement interval of 5% and 0, 0.125, 0.25, 0.5 and 0.75% were the various percentage of CC fibers addition to the mortar. The addition of CC fibers increased the flexural strength but reduced the compressive strength [106].

RESULTS AND DISCUSSION

Limiting the utilization of cement in construction industry, SCM plays an active role. Sources of SCM are numerous but those evolving from biomass will have an impact on the future. Presently, 81% of the global energy needs are fulfilled by fossil fuels whereas 19% is contributed by renewable energy sources [107]. The agricultural biomass designated as carbon neutral possess a major drawback, posing a problem of ash disposal. The ash on an average account for 20% of the biomass waste on a dry basis. This ash is a potential resource for the construction industry as SCM if properly calcinated. The major parameters that influence the characteristic of ash are temperature, retention time, heating rate, cooling method and combustion technology. These parameters significantly alter the morphology, physical and chemical aspects of resulting ash.

Calcination Parameters

The first and foremost calcination parameter is temperature. A control over temperature modulates the chemical reactions to yield intended quality of ash. But it is not solely dependent on temperature. Retention of biomass for a particular time inside combustion chamber at desired temperature yield fine quality of ash. Whenever the temperature is kept less than 600°C a greater extent of retention time is required. If retention time is lower, char combustion (oxidation of carbon) remains incomplete. Because at lower temperatures, oxygen diffusion rate is greater than rate of chemical reaction. Since the chemical reaction pace is slower, the complete oxidation of char requires greater retention period. The diffusion of oxygen to the inside of char particles favors oxidation from inside resulting into ash with lower densities. A rise in temperature retards the oxygen diffusion and increases the chemical reactivity. Whenever calcination temperature goes beyond 900°C, retention time required is less than an hour. Due to higher chemical reactivity, oxygen diffusion to the inside of particle is not likely to happen. Instead, as oxygen from the bulk gases reaches particle surface instantly starts oxidizing the surface carbon thus reducing the particle size. Ashes that are burnt at higher temperature are likely to have greater densities than those at lower temperature.

The other factors i.e. heating rate and cooling method though secondary, play a vital role in enhancing the quality of ash. A higher heating rate escalates the carbon content inside the ash due to the presence of K_2O impurity. For higher heating rates, desired temperature is attained faster which favors early dissociation of K_2O into elemental K^+ ion. The early released K^+ ion gets absorbed on the surface of silica particle and cause surface melting which traps carbon inside and results in high carbon ash [108]. In the process of slow heating rate enough time is available for thermal decomposition of carbon before the dissociation of K_2O into elemental potassium. Since, the formation of elemental potassium takes places at later stages, greater time span is available for carbon removal. This indicates that a slow heating rate has a higher efficiency of carbon removal than higher heating rate.

The rapid cooling method promotes the retention of amorphous silica phases whereas slow cooling results into higher percentage of crystallinity inside the ash. In the slow cooling process, the dissociated K^+ ion which is already present inside the ash will cause melting of the particle surface on which it is deposited. This promotes the crystallite growth of the particle. A rise in the crystallite size increases the crystallinity and lowers the pozzolanic reactivity inside the ash. In case of rapidly cooled ashes, the drop in temperature occurs very quickly, which stops the surface melting of the particle due to deposited elemental potassium.

This hinders the crystallite growth and results into amorphous ash with high pozzolanic activity [109].

Mechanical Properties of Concrete

Effect of calcination parameters

Calcination parameters influence mechanical characteristics of concrete. Various investigation outcomes revealed 500-700°C as optimum calcination temperature. Such ash when integrated with concrete yields greatest mechanical performance. At higher temperature (>700°C), crystallization of amorphous phases occurs which lowers mechanical performance of concrete because crystalline phases do not react to form C-S-H gel. Even high temperatures yields amorphous silica, but at lower retention time. The mechanical performance of concrete with BMA containing equal quantum of amorphous phases produced at temperature < 700°C should conceptually be greater than BMA produced at temperature >700°C. Because temperature < 700°C favors decarbonization to the inside of particle which enhances the reactivity due to greater amount of oxygen diffusion. For a retention time of 2-5 hours in low temperature region, ash contains minimum carbon and crystalline phases which yields optimum mechanical performance of concrete. Heating rate less than 10°C/min and utilizing rapid cooling regime leads to ash with low carbon content and crystalline phases respectively. Concrete developed incorporating such ash shows greater mechanical performance.

Effect of varying BMA content

Researchers have used different parameters for thermal treatment of RHA. This has resulted into variable cement replacement percentage within the cement mortar/concrete mixes. From the past studies it is observed that, in development UHPC and achieving highest compressive strength Huang et.al. utilized 25% RHA whereas Farid et.al. could utilize only 3% RHA. Such results are not only observed in case of UHPC but also for other types of concrete. This variability in percentage replacement is due to the difference in thermal treatment parameters and neglecting parameter like heating rate and cooling method during the process of treatment. Therefore, it is very crucial to consider and control all the parameters of thermal treatment within the optimum range. This would ensure a good pozzolanic behavior of BMA and could bring the percentage consumption of BMA within a certain range.

Synergistic effect of biomass ash with different fibers

The addition of thermally treated RHA along with different fiber types improves the mechanical performance of the cement-based matrix. The addition (cement replacement) of RHA (without inclusion of fibers) has greater improvement on the mechanical performance of cement-based matrix than the addition of various fiber types (without inclusion of RHA). RHA induces a performance increase in the range of 5 – 20%, whereas the fibers increase the performance between 2 – 8%. The reactive silica present in RHA interacts with calcium hydroxide present in cement matrix. Their interaction in the presence of pore solution produces additional C-S-H gel which enhance the pore structure within the matrix, especially the interfacial transition zone (ITZ) and the improved pore structure results in an increase in strength of hardened matrix. On the other hand, the addition of different fibers along with RHA in the cement matrix elevates the strength over and above the cement + RHA matrix. The random dispersion and interfacial bond between the fibers and cement matrix enhances the strength, toughness, ductility, crack and fatigue resistance of the matrix. It is also seen that, the addition of RHA and fibers lowers the workability of cement matrix, whereas the setting time is delayed due to RHA and not because of fibers.

CONCLUSION

Biomass is one of the most promising fuel sources for energy generation. Ash evolving out from combustion of biomass can potentially be utilized in construction industry. But utilization of ash is dependent on chemical, physical and morphological characteristics. The chemical and morphological features are greatly influenced by calcination. Parameters of calcination regulated at optimum level render ash with maximum reactivity for substitution as supplementary cementitious material in concrete and mortar.

a. Parameters of calcination influencing properties of ash were reviewed. These parameters govern the phase and content of carbon in ash.
b. Temperature and retention time can be considered as primary and heating rate and cooling method as secondary parameters. Because unless the primary parameters are regulated at optimum level till then the effect of secondary parameters cannot be seen.
c. Temperature of 500-700°C is the optimum range for calcination of biomass, raising temperature beyond 700°C brings down the quantum of amorphous phases in ash.
d. Within the optimum temperature range, the retention should be maintained between 2 – 5 hours, heating rate should be less than 10 ^{o}C/ min and the ash should be cooled rapidly to obtain high pozzolanic activity in ash.
e. A variation of these parameters above and below optimum level affects the mechanical performance of concrete because of change in chemical and morphological characteristics.
f. Fluidized bed combustor is an efficient technology of biomass fuel combustion compared to moving grate firing system when the quality of ash is concerned. Even suspension fired burner are efficient but the problems associated with fuel quality, demand preprocessing/preconditioning of fuel, complicated the working and increase maintenance of system tags it as a low popular system.
g. Not all the available biomass ashes could be classified as supplementary cementitious material. Therefore, it is

very important to check with the criteria of SCM mentioned in IS 3812: 2013 and ASTM C618.

h. The attributes of ash and concrete incorporating the ash is available over a wide range of temperature but the effect of retention time, heating rate and cooling method are not available at high temperature values.

i. Though heating rate and cooling method are secondary parameters but influence characteristic of ash. Effect of varying heating rate and cooling method at low, optimum and high calcination temperature and retention time on mechanical properties of concrete is an area with wide scope of research as per the review.

Practical Application

The biomass ash to be utilized as supplementary cementing material should satisfy the chemical composition criteria mentioned IS 3812: 2013. Generally, the biomass ash such as rice husk ash, sugarcane bagasse ash, wheat straw ash, bamboo leaf ash etc. contains considerable carbon content which may be detrimental to the strength, durability and other vital parameters of cement-based matrix. Moreover, the carbon cover over the ash particles inhibits the reactivity of ash. Therefore, thermal treatment of biomass ash is necessary for carbon removal. When any of the identified biomass ash containing substantial amount of carbon is to be used in construction as cement replacement, it should first undergo a thermal treatment process with the optimum control of thermal treatment parameters. The identified thermal treatment parameters are temperature, retention time, heating rate and cooling method. The temperature, retention time and heating rate should be regulated between 500 – 700 ºC, 1 – 3 hours and 1 – 10 ºC/minute, followed by a rapid cooling method to stop the crystallite growth and conversion of amorphous phases into crystalline phases. The practitioners should note that maintaining high treatment temperature (close to 700 ºC but not greater than 700 ºC) requires less retention time, whereas maintaining lower temperature (close to 500 ºC but not less than 500 ºC) demands higher retention time within the stated optimum range. When such ashes are used by the practitioners in construction industry the cement substitution can take place between 5 – 40% depending on the required strength of concrete. The review reveals that, low grade concrete (required strength < 25 MPa) can substitute cement with biomass ash as high as 30 – 40% whereas in high strength concrete (required strength > 60 MPa) the cement is replaced between 5 – 10%. The replacement of cement with biomass ash not only lowers the construction cost but also decelerates the rate of cement requirement which eventually lowers the pace of raw material required for cement production. The decelerated pace of cement requirement will bring down the carbon emissions, conserve natural resources thereby promoting sustainability.

AUTHORSHIP CONTRIBUTIONS

Authors equally contributed to this work.

DATA AVAILABILITY STATEMENT

The authors confirm that the data that supports the findings of this study are available within the article. Raw data that support the finding of this study are available from the corresponding author, upon reasonable request.

CONFLICT OF INTEREST

The author declared no potential conflicts of interest with respect to the research, authorship, and/or publication of this article.

ETHICS

There are no ethical issues with the publication of this manuscript.

STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE

Artificial intelligence was not used in the preparation of the article.

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