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EVALUATING THE PROCESSING OF ALUM SLUDGE AS A POZZOLANIC MATERIAL: A REVIEW

Abstract. Alum sludge (AS) is an inevitable by-product generated during water purification and wastewater treatment plant when aluminium salts are used during the coagulation process. The study primarily aimed at comprehensive review of the physical, chemical and microstructure properties of alum sludge, assessing its suitability to act as a pozzolanic material in cement as a partial replacement for ordinary cementitious materials. AS has been proven to have high amount of silicon dioxide (SiO_2), iron oxide (Fe_2O_3) and aluminium oxide (Al_2O_3) from XRD analysis. Scanning Electron Microscopic (SEM) was conducted to fully understand the morphological structure of AS. Various analytical techniques are implemented to assess the pozzolanic potential of alum sludge. Comparative studies have highlighted the use of alum sludge in concrete which shows a favourable outcome in terms of the mechanical strength including compressive strength, flexural strength and durability. The findings in the present studies suggest that with a proper treatment and processing, alum sludge can be a viable and eco-friendly alternative to conventional pozzolanic materials. The future challenges of the process in the development of an effective alum sludge were discussed.

Keywords: Cement; chemical properties; physical properties; Alum sludge; microstructure properties

1. Introduction

The huge demand for cement production is increasing greenhouse gas emissions and responsible for 8% of global warming issue [1,2]. Mainly in Malaysia, the demand for cement production has increased from 19.86 million metric tons in 2022 to 23 million metric ton in 2023. In 2023, China has recorded to be the highest country with the most cement production globally with an estimation of 2.1 billion metric tons in 2022 followed by India with 370 million metric tons [3]. Therefore, the investigation on utilizing potential material for cement replacement is necessary. Therefore, various pozzolanic materials are explored aggressively to replace part of cement to minimize this environmental issue [4,5]. Recycling industrial waste as a partial substitute in concrete production could contribute to sustainability in industry and reducing environmental impact of increasing waste disposal [6]. Varieties of waste materials have been explored such as fly ash, coal bottom ash [7], sugarcane bagasse ash [8,9] and including alum sludge. Alum sludge can be considered as a pozzolanic material due to its chemical composition that is quite similar to conventional cement. Numerous of study shown the possibility of utilizing alum sludge for brick, cement or other building materials [10-11].

Alum sludge (AS) or hydro solids are a wastewater residual that is generated during the process of drinking water treatment [12]. The swift growth of the populations is making the demand for clean water drinking water increase rapidly [13]. The drinking water treatment facilities usually mix iron-based chemicals into the water during cleaning process [14]. The coagulant formed contains a variety of contaminants such as bacteria and particles. Generally, alum sludge contains aluminum sulphate because it is the common coagulant substances used in drinking water treatment plants during coagulation-flocculation process [15]. The mass production of this material caused environmental pollution due to poor industrial waste management.

The common way to dispose of alum sludge is through landfills. However, this method is not recommended nowadays due to limitation of landfills spaces and the increasing landfills management cost [16,17]. Netherland has spent more than 30 to 40 million pound per year for sludge disposal, and it is predicted that the cost will be doubled by the next decades [18]. Meanwhile in United Kingdom, they have spent estimated of more than 5.5 million pound in 2000 [19]. In fact, from the perspective of economic concerns, the disposal of this waste material has been expected to rise considerably in the enforcement of taxation or legal prevention of landfilling. In Malaysia, the production of

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alum sludge per year can reach up to 2.0 million metric tons and it is illegal to dispose the alum sludge downstream of water [20]. This issue also happened in other countries as well, such as China, Spain, Japan etc. which produced over 7 million tons of alum sludge annually [21-23]. There is a hot debate on water pollution due to toxic aluminum ion concentration (Al^{3+}) in the water which noted the concentration mixed of $AlOH^{2+}$ is hazard towards human health as well as the animals [19]. Several studies stated that alum sludge can be utilized in any industry such as agricultural, ceramic production and supplementary material in cement. However, there is no specific global standard to utilize alum sludge as cement replacement material.

The physical and chemical properties of alum sludge might vary depending on the amount of coagulant added in the wastewater, suspended impurities and particles at the bottom of the collecting tank. As the present of impurities could affect the structural integrity of concrete system, hence, proper treatment is necessary to ensure that this sludge is safe to be applied in concrete [24]. Besides, the quality of water sources and the process of the treatment also contributed to the diversity of the properties [13]. A material that can be considered as a pozzolanic material should have the main components, aluminum and silicates compound. In alum sludge, there are a lot of useful chemical compositions such as high content of aluminum oxide Al_2O_3, silicon dioxide SiO_2, iron oxide Fe_2O_3 and calcium dioxide [11]. This chemical content is similar to conventional cement used in construction materials which helps in producing extra binding phases and greater mechanical qualities in concrete

2. Production of alum sludge

National Water Services Commission mentioned that Malaysia's water treatment plant generated in average of 5500 metric tons of alum sludge per day and around 2 million tons annually [15]. This heterogenous solid waste contains high concentration of aluminium hydroxide and certain impurities when suspended solids, organic matter, liquefy colloids and microorganism in raw water being mixed [25]. The recurrent version of disposal method for AS is through landfills which has caused the increasing of cost in drinking water treatment industry due to limited spaces and transportation [26]. The process to treat drinking water consists of two main stages and another one additional stage if required [27]. Both stages capable to eliminate different percentages of suspended solid or AS. Up to 60% and 85% of solid can be removed in stage one (sedimentation process) and stage two (filtration process) respectively. Meanwhile, for a contaminated water, stage three will be required, and it could eliminate up to 99% of suspended solid.

Alum sludge is generated during the coagulation-flocculation process where aluminium salts is added into water to act as coagulant. Aluminium hydroxide flocs precipitate and pick up pollutants and any precipitation from the water, forming by-product called alum sludge [28]. General steps to produce the sludge after coagulation are sedimentation process and filtration process. Fig. 1 below shows the graph indicated the average of alum sludge production by drinking water treatment process annually extracted from [21-23,25].

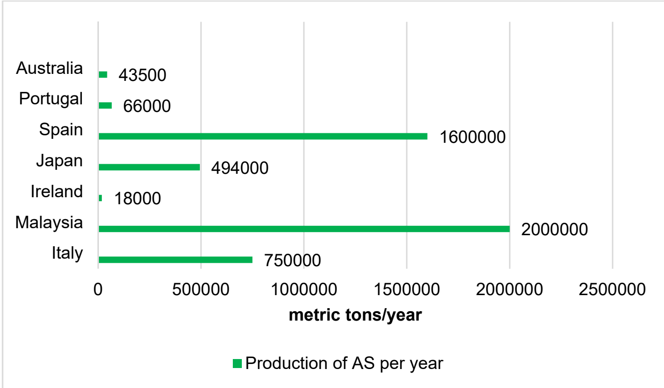

Fig. 1. Average production of AS per year in different countries

3. Process of treating water in water treatment plant

The process of water treatment starts with collecting raw water from any water sources such as riverbanks. The water then will be transported to any nearby water treatment plant. During screening process, huge debris is being removed such as wood, rags and any bulky object that could stuck in the narrow grit chamber [27]. The narrow grit chamber will slow down the flow of water and let rough grits such as gravel, sand, coffee grounds and eggshells to settle out of the water since it can affect other functionally of the equipment on next stages. Then, in the next stage, which is the coagulation and flocculation process, aluminium sulphate is added to neutralize charges on particles and clump the suspended particles together as shown in Fig. 2. This step is crucial as it is the main reason of alum sludge production. The water is mixed slowly to allow particles to collide and sticks to each other which form large solids called flocs. Then during the sedimentation process, the water that contain suspended solid will be keep for more than two hours for gravity sedimentation. The solids obtained from sedimentation tank is called primary sludge. The primary sludge generally has strong odour as it contains a lot of organic material and has high solids content. The solids contain organic and inorganic solids, grease, grit and small debris and pumped out for removal. The texture of primary sludge is denser and coarser as it contains large flocs.

The leftover solid that escape from sedimentation process will be removed during filtration process. During filtration process, water passes through sand, gravel or multimedia filters to remove remaining fine particles and flocs. The remaining solids called as secondary sludge. Secondary sludge might have slightly odour but not as strong as the primary sludge and waterier as most of suspended solids have been removed before. The texture is finer compared to primary sludge due to the filter capture. Both sludges are chemically the same even though they were extracted from different stages. Before the water is storage for distribution

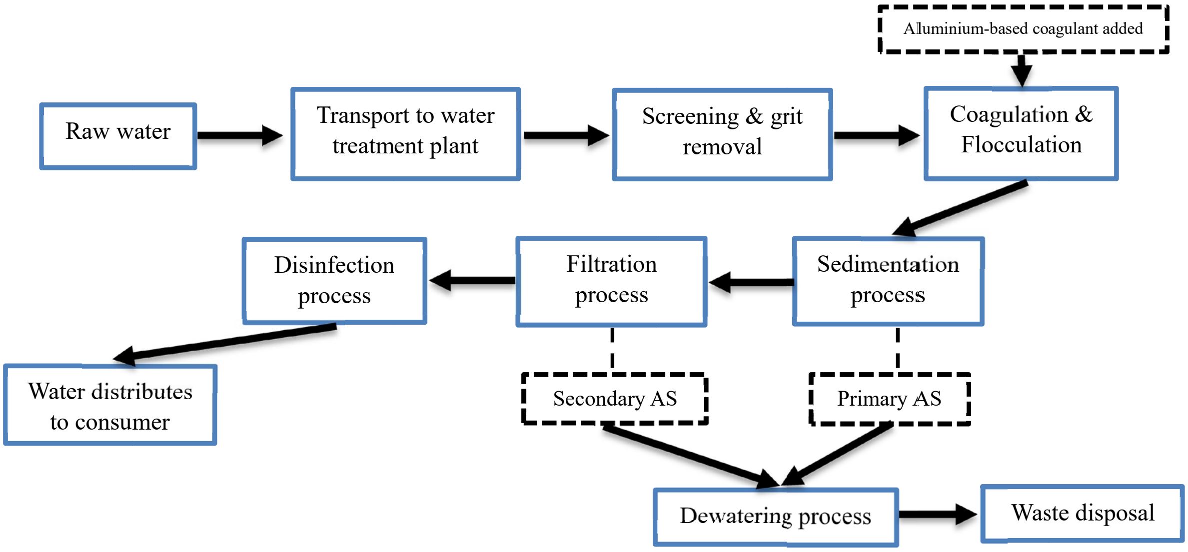

Fig. 2. The conventional treatment of drinking water process [35-37]

to the consumers, the water is going through disinfection process. Chlorine is commonly used during this stage which mainly to kill pathogens and to ensure that the water is safe to be consumed. All alum sludge (primary and secondary sludge) collected from the sedimentation and filtration process will undergo dewatering process to reduce its volume and weight by several methods. The methods are belt filter presses, centrifuges, screw presses and drying beds. Then, the alum sludge produced will be transported to landfills or waterbody to be dumped.

4. Physical and chemical properties of alum sludge

Physical properties of alum sludge depending on the water resources, treatment process, quality of raw water and the method of transporting the material [13]. In alum sludge, the physical properties is determine by its moisture content, the density if alum sludge, particle size and the porosity of the material. It is significant to understand the properties as it might give impact towards potential uses. The specific gravity and water absorption capacity of alum sludge were evaluated according to the standard procedures, ASTM D 854-00. The particle size of alum sludge depending on the number of cycle of milling machine and the efficiency of the equipment. Raw alum sludge has average particle size distribution of 100 μm [30]. However, in cementitious replacement materials, the size should be passing 45 μm which the size of the ordinary cement. TABLE 1 shows the physical properties of alum sludge from several research.

Generally, based on ASTM618, the acceptable density of the material to be used as replacement not exceed 5%. A high-density AS has more tightly packed particles which called "packing effect" [34]. In the study by [13], they notified that the bulk density of specimen produced by incorporating alum sludge with 15%-30% of all types, raw AS, thermally activated AS and pulverized AS produces lower bulk density than normal concrete.

Other study by [32] mentioned that pulse velocity is relatively related with density and elastic properties of the material. The researchers also mentioned that a lower density of AS usually contains high porosity which providing greater surface area and giving chance for reactivity to occur between AS and cement. Aside from the physical properties, chemical properties of alum sludge based on the amount of the coagulant added during the water treatment process [16]. These characteristics also influenced by the impurities contained in the water discharged into the resources. In Malaysia, the heavy metals contain in sludge is within the safe limits [35]. High content of silica and aluminum oxides in the alum sludge come from the added coagulants which contain aluminum sulphate and trivalent iron salts [36].

TABLE 1

Physical properties of alum sludge from previous study

Author & Year	Density (g/cm^3)	Specific surface area (m^2/kg)	Specific gravity	Average particle size (μm)	Moisture content (%)	Colour	Source AS
[13]	2.24	326	—	57.502	2.42	Milky white	Pulau Indah, Malaysia
[31]	1.30	—	2.68	—	—	Light gray	Dakahlia, Egypt
[26]	—	—	2.34	—	0.85	—	Kurdistan, Iraq
[32]	—	650	2.38	24.40	—	Light brown	Putrajaya, Malaysia
[16]	2.20	—	—	7.51	—	—	York, United Kingdom
[33]	2.8	423	2.3	75.0	0.5	—	South Australia
[22]	—	1100	2.57	11.77	—	Orange	Putrajaya, Malaysia

TABLE 1 shows the physical properties of alum sludge. Based on ASTM 618 [37], the moisture content in alum sludge for a natural pozzolan cannot exceed 3%. The average particle size of alum sludge should be less than 75 µm as it could affect the reactivity for pozzolanic reactions.

TABLE 2 show the overall chemical composition existed in alum sludge, The main chemical elements are silicon dioxide (SiO_2), aluminium oxide (Al_2O_3) and iron oxide (Fe_2O_3) [13,14,26,33]. These three element must exceed 70% of the total percentages to pass as a pozzolanic material based on ASTM 618. However, precise amount of oxides required depends on the specific application and desired properties of the material.

5. Microstructure properties of alum sludge

Microstructural analysis of alum sludge is important aspects to be considered when it comes to getting an insight regarding the materials' qualities and potential applications that it can be utilized within construction sector. Liu [33] investigated the microstructure features of alum sludge ash. X-ray diffraction (XRD) test was conducted to analyse the mineralogy compositions of the mortar after exposure to 550°C and 800°C. The mortar paste was ground less than 36 µm, and the sample was scanned using an Empyrean diffractometer (Malvern PANalytical) with Cu Kα radiation at 40 kV and 40 mA. Other testing also was conducted such as thermogravimetric analysis (TGA) and derivative thermogravimetric analysis (DTG) curves to identify the thermal stability and composition of the material.

In another study by [38], XRD method was also be used to analyse OPC and water treatment sludge for its mineralogical crystalline phases and compound composition. This study used a diffractometer with monochromatic Cu-Kß source, curved graphite including a single-crystal (1D) chromator set up at 40 kV, 50 mA, 15°/min, with a Hypix-400 horizontal detector. The samples were scanned over 2ϴ range of 5°-9° at scan rate of 1/2° per minute with 0.001° increment. The result of XRD testing can be seen from Fig. 3 below.

In Fig. 3, the highest peak reach at 26°-28° 2ϴ. This is likely due to the corresponding toward quartz which in this case sulphate oxide (SiO_2). It is a common crystalline existed in alum sludge and non-reactive mineral phase. The multiple short sharp peaks across the spectrum indicate the presence of well-crystallized phases such as iron oxides or other minerals such as kaolinite, gibbsite or aluminium hydroxide. From the result, this sample shows a low pozzolanic reactivity. However, it can be improved by thermally or chemically treated proven by the research conducted by [30,32].

TABLE 2

Chemical properties of alum sludge from previous study

Ref.	SiO_4	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_4^{2-}	Cl^-	Na_2O	K_2O	TiO_2	MnO	CuO
[33]	6.7	—	194.49	5.2	5.18	1.59	8.86	11.39	—	—	—	—	—
[34]	—	10.28	44.24	2.51	2.50	0.35	—	—	0.15	0.43	0.16	0.15	—
[35]	—	31.11	47.68	4.94	4.32	0.96	3.39	—	0.19	0.97	—	—	0.29
[36]	—	42.32	35.03	5.18	0.13	0.30	—	—	0.10	1.87	—	—	—
[37]	—	42.38	35.03	4.94	0.13	0.29	—	—	0.03	—	—	—	—
[38]	—	37.48	28.84	9.92	3.1	0.64	—	—	0.30	0.75	1.28	—	—
[39]	—	36.38	19.74	2.34	2.83	0.24	—	—	0.14	0.43	0.18	—	—
[40]	—	44.21	16.47	4.12	4.62	0.74	—	0.018	0.61	0.31	—	—	—
[17]	—	36.29	27.92	5.33	3.77	1.12	—	—	1.31	1.81	—	—	—
[41]	—	42.09	19.73	5.57	8.21	1.34	—	—	0.52	1.74	0.59	0.03	—

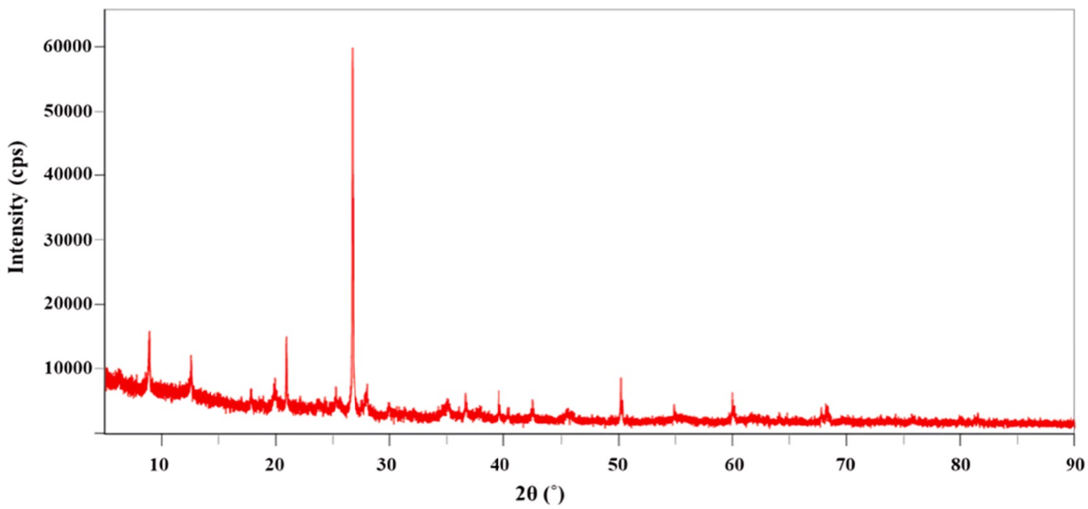

Fig. 3. X-ray diffraction (XRD) pattern for modified water treatment sludge [45]

Based on [10], Scanning Electron Microscopic (SEM) is necessary to determine the filler effect of the replacement material on concrete. [11] mentioned is the research on the influence of thermally activated alum sludge ash that from SEM result, it can be concluded that alum sludge is quite similar to an amorphous structure which the particles exhibit a rough and compact texture. The rough and irregular surfaces provide more area for hydration process and provide better mechanical interlocking which help the material to bond better with cement paste. Fig. 4 shows the SEM image of raw alum sludge.

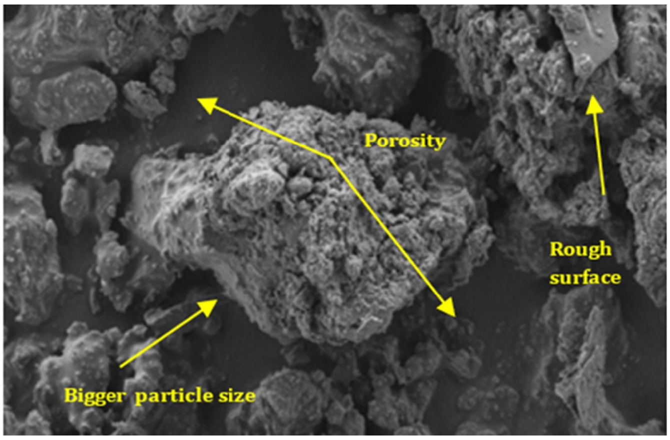

Fig. 4. SEM image of raw alum sludge ash [47]

The magnification used must at high range to properly and clearly examine the shape, texture and particle distribution [38]. It is important to check every aspect as mentioned because the particle size and porosity of the material might affect the rate of pozzolanic reaction and workability. Jia [39] also conduct a microstructure evaluation of mortar based on alum sludge ash which for this case the research in determining the essence understanding that exist between the microstructure and the mechanical performance in this instance. Both samples were tested after passing 90 days of curing period. In Fig. 4 shows SEM images of mortar with 0% of alum sludge and mortar with 30% of alum sludge as cement replacement.

Based on the images in Fig. 5, it is proven that the pozzolanic reaction occurred. As can be seen from the normal sample, the irregular shaped resemble like a cotton gel is actually a C-S-H gel, which acts like glue in hardened cement. The hexagon-shaped crystal is calcium hydroxide (CH) which a common byproduct of cement hydration. Another common material in cement that can be seen is the needle-like crystals which is ettringite which is another product for hydration. In the sample with 30% of alum sludge replacement, there are a lot of ettringite compared to the normal sample. CH crystals were used up in the pozzolanic reaction, therefore, only few CH crystals can be seen from the images. Hence, in this research, alum sludge is proven as a good pozzolan materials as SEM images proves

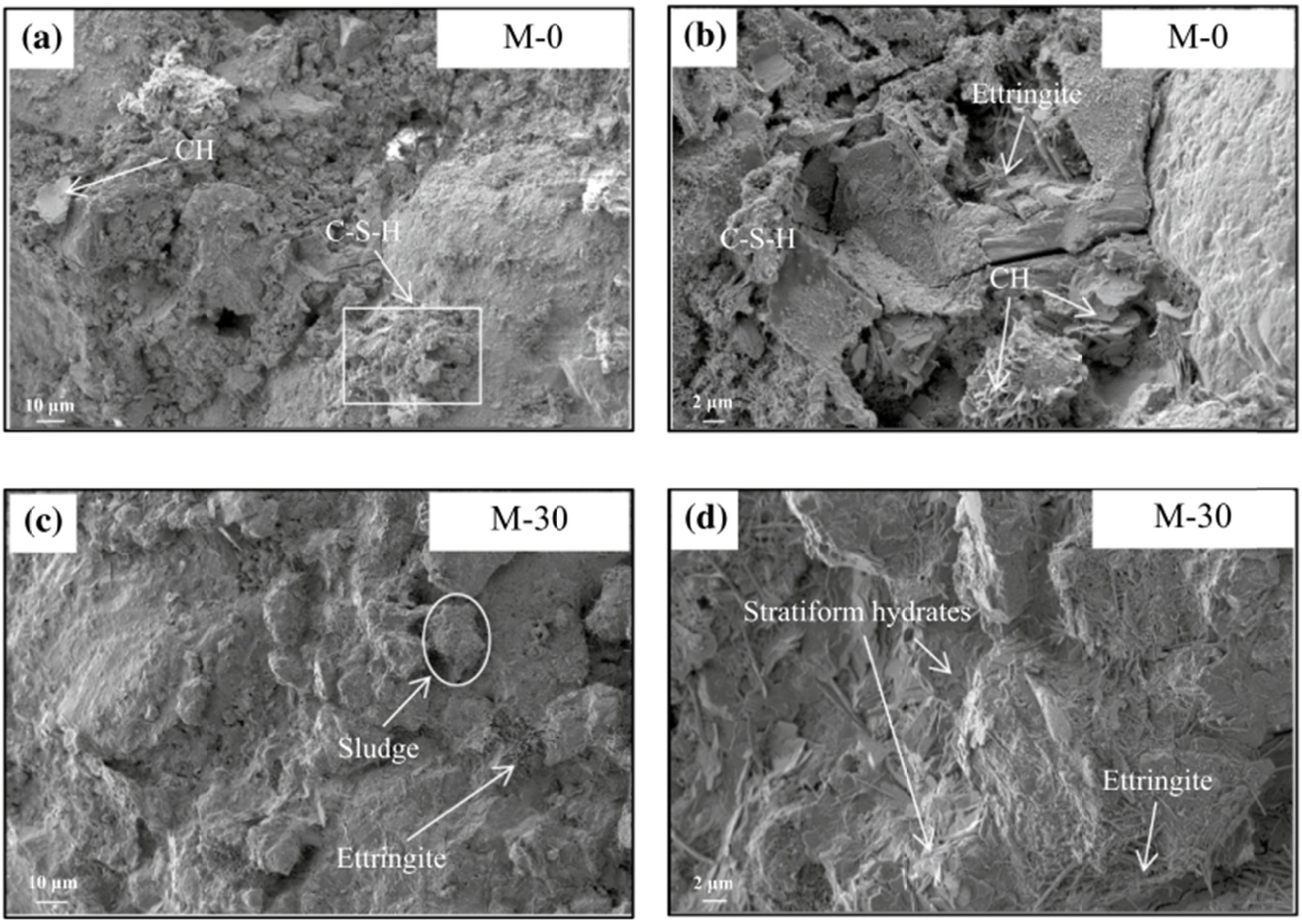

Fig. 5. (a) and (b) Mortar with 0% AS, (c) and (d) Mortar with 30% replacement of AS

that the sludge is reacting in the mix and can help in improving strength over time.

6. Pozzolanic reaction of alum sludge

In the sphere of ecologically natural building materials, the pozzolanic reaction of alum sludge is vital because other high-alumina alum substitutes are also employed in the context of cements. Alum sludge is called on to provoke this reaction as a pozzolanic material. Alum sludge is categorized as a raw natural pozzolans which including in class N. Generally, criteria of a good pozzolan are when the material meets the standard requirements as stated in ASTM 618. A great pozzolan material is when the total of chemical composition of silicon dioxide (SiO_2), aluminium oxide (Al_2O_3) and iron oxide (Fe_2O_3) exceed 70% of the chemical content. In the presence of water, the material alum sludge interacts with calcium hydroxide as Ca $(OH)_2$, resulting in the generation of additional cementing compounds which is calcium silicate hydration (C-S-H) [40]. Frattini test, strength activity index test (SAI) and calcium hydroxide consumption test are the common tests to evaluate pozzolanic activity of sludge [41]. Chang [41] research proved that alum sludge achieved the highest pozzolan activity when it is calcined at 800°C and 900°C based on the result shows from Frattini test corresponding to SAI test result.

Mohamed [42] researched environmentally friendly and low-cost alum sludge by-products. The study's results show that alum sludge has considerable capacity when replacing Portland cement. Investigating substitute materials since they reduce the construction industry's influence on the environment and double the effectiveness of construction. Kaish [32] reviewed about the significant proof from some research that mentioned alum sludge can be used partially as cement replacement after thermally activated. Owaid [43] investigated the used of alum sludge the use of sludge from water treatment plant. Chemical analysis was conducted to identify the chemical composition. Other strength tests such as flexural test, tensile test and compressive test of the concrete were evaluated and the result shows 6% of alum sludge replacement had higher strength with presence of ferric oxide alumina and silica in alum sludge and ordinary cement.

7. Mechanical properties of alum sludge in concrete

The material's mechanical qualities are the sigificant factors to be considered. Kaish [32] studied the mechanical properties of concrete when a certain proportion of fine aggregate was replaced by alum sludge. Based on the results of this experiment, the authors experimenting on the effect of this substitution on the compressive and flexural strength and durability of concrete mixtures. It was mentioned in the article that the optimum replacement content of fine aggregate that has been oven-dried and heated at 300°C as a fine aggregate replacement was 10%. The strength and durability properties of the concrete yielded better at this replacement meanwhile 15% yield a poor concrete properties. Therefore, having an accurate understanding of these mechanical qualities is essential to be certain that the concrete structures produced will fulfil the necessary performance criteria.

Experimental investigations on the concrete specimen with partial alum sludge as cement replacement has been conducted in few studies to evaluate the properties of hardened concrete. Compressive strength can be defined as the load which causes the failure of standard specimen divided by area of cross section in uni-axial compression under given rate of loading [44]. Suchand [44] conducted a compressive strength on the concrete with different percentages of AS (0%, 5%, 10%, 15% and 20%). The result as shown in the Fig. 6 below shows only minimal variation by replacement of cement with acceptable standard strength at 15% replacement of AS in the concrete before major reduction of strength occurred.

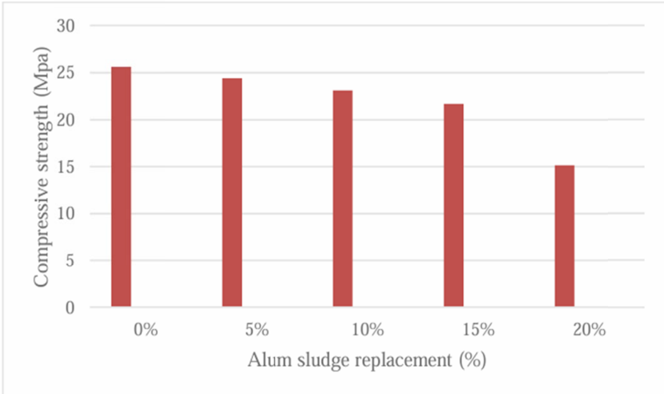

Fig. 6. Compressive strength of concrete with AS replacement [49]

In other study by [17] tested compressive strength by using concrete cubes with 2000 kN compression machine. In this study, the compressive strength of controlled mix with 2% of superplasticizer (SP) (CTSP2) was at 41.43 MPa meanwhile, the compressive strength of the sample labelled as AS4SP2 which indicating the sample has 4% of alum sludge as cement replacement with addition of 2% superplatizer (SP) was at 30.83 MPa on 28 days. Eventhough the AS4SP have lower strength than CTSP2, the strength is still within the limits as per the definition of normal concrete which was 20 N/mm^2. Therefore, in this study that is the optimum replacement of cement is 4% with addition of 2% SP.

The result for compressive strength could be slightly similar to flexural strength. The flexural strength of the sample with 2% alum slude replacement with 0 SP (AS2SP0) decreased more than 50% compared to the control mix (CT) at 28 days. As can be seen from the graph in Fig. 7, the flexural strength for all alum sludge sample with SP (AS2SP2, AS4SP2, AS6SP2 and AS8SP2) have better result compared to AS2SP0. Therefore, it is important to add SP in the production of concrete to improve cement hydration as well as improving the overall performance of the concrete. Hence, the strength of concrete specimen with AS replacement of cement could be varies depending on the treatment process of AS and the concrete production.

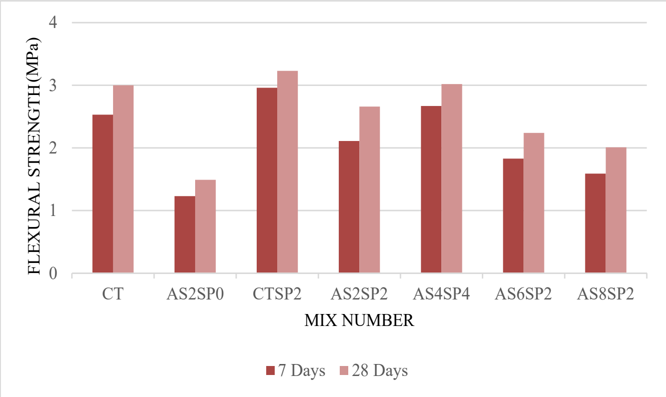

Fig. 7. Flexural strength of concrete in different conditions [54]

To examine the durability, Suchand [44] tested the durability of the concrete specimens by conducting acid attack test for 0% replacement and 15% replacement of AS specimen. After 7 days of curing, the samples were kept in atmosphere for 2 days and weighed. Then, both were kept in 5% of sulphuric acid for 60 days and kept in atmosphere for 2 days again before weighed. In this test, the percentages of weight loss for both cubes were 4.46% and 5.70% respectively. The loss of weight is because when concrete is exposed to acidic solutions, it can degrade over time as the solutions chemically breakdown and eventually dissolve components of the concrete matrix. Moreover, the reaction between calcium hydroxide (CH) and C-S-H gel with acid will produces gypsum ($CASO_4 \cdot 2H_20$) which can expand and causing internal crack of the concrete which affecting the loss of binding and material integrity.

In other study, Owaid [43] evaluated the cube specimens on the 28 days of curing periods. The specimens were immersed in 5% sodium chloride (NaCl) solution. This test was performed according to ASTM C1012. On the test day, all concrete specimens have no visualized impact or damaged on the surface after 90 days and 180 days. All specimens were evaluated using compressive strength test. The ternary blended binder (TBB) concrete had better compressive strength than binary blend (BB) cement with the same amount of replacement levels. This can be justified as the mixes of cement in TBB combining three cementitious materials such as palm oil fuel ash (POFA), silica fume (SF), ground granulated blast furnace slag (GGBS) including activated alum sludge ash (AASA). All these materials are good pozzolans. Meanwhile, the binary blended cement (BB) only combined two of those cementitious materials. Therefore, each of the materials combined in TBB complement each other to improve the strength and reducing the porosity of the concrete as they have different reactivity rate and compositions. Besides, combining three materials as cement replacement in concrete make the concrete matrix denser which also enhancing durability. Moreover, every material has different sizes which helps in mechanical interlock between particles in the concrete and improve packing density. In this study, concrete containing 6% SF, 20% GGBS and 15% POFA showed better resistance to sodium chloride attack. However, all TBB concrete with the combination of AASA with other pozzolan materials cured under chloride solution had better compressive strength.

8. Conclusion and recommendations

In this review paper shows the production of alum sludge and the evaluation of alum sludge for its pozzolanic reactivity when the material is being used as replacement in concrete. From the testing conducted, it is proven that alum sludge passed the requirements for natural pozzolan materials. Some study indicates that the thermally treated alum sludge shows great performance compared to the untreated alum sludge. Besides, in some research that there is slightly reduction of mechanical strength of concrete with partial AS replacement compared to the conventional Ordinary Portland Cement concrete but still within the acceptable range as a normal concrete which at 20 N/mm^2. It can be concluded that the average optimum replacement of alum sludge is at 15%.

Treating alum sludge in various way is recommended in the future research to identify another method to enhance pozzolanic reactivity other than calcinate the alum sludge or doing chemical treatment. Hence, the research could help in maximize the amount of alum sludge used in the concrete production as well as minimizing industrial waste dumping into landfills. Besides, in implementing this study in real life industry might have challenges as there is no standardization or regulatory guidelines on alum sludge use in concrete which including application protocols and handling techniques.

Acknowledgement

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