

# RELIABILITY OF ALKALI-ACTIVATED AND PORTLAND CEMENT MORTAR UNDER COMPRESSIVE TEST BY ACOUSTIC EMISSION

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**ABSTRACT:** Alkali-activated materials or geopolymer technology is one of the material innovations that can provide several benefits by reducing the use of Portland cement. The source of precursor materials come from industrial by-products. This study investigated the reliability failure of seven-variation of alkali-activated fly ash/slag/micro-silica mortar (AAM) and one-type of Portland Cement (PC) mortar. Compressive strength test accompanied by acoustic emission is used to characterize cylindrical mortar specimens. Examination of fracture distribution according to stress level until final failure was then performed. The compressive strength of four-type of alkali-activated mortar AAM (AAM-IV, V, VI, and VII) at 14 and 28 days shows a slight strength increase, 61.9 to 63.6 MPa, 62.3 to 65.6 MPa, 64.8 to 68.3 MPa and 63.1 to 63.6 MPa, respectively. The slight increase of AAM compressive strength is caused by high early strength achieved due to the replacement of more than 40 % of fly ash with GGBFS. The presence of more CaO in the AAM mixture accelerates the reaction in early age. By contrast, PC mortar shows significantly strength increases from 55.2 MPa to 69.5 MPa during the same period. Amplitude filters greater than (50dB, 60dB, 70dB and 80 dB) is utilized to investigate the reliability of compressive strength of the mortar by acoustic emission. It was found that filter greater than 60dB is the most suitable filter. Alkali-activated mortars which contain raw material of 42.5% fly ash, 42.5% GGBFS, and 15% micro-silica has the lowest reliability of failure than those of other mortars whereas Portland cement mortar shows the highest reliability of failure than other alkali-activated mortars.

*Keywords: Acoustic Emissions, Alkali-activated materials, Geopolymer, Reliability, Fly Ash, Slag*

## 1. INTRODUCTION

Geopolymer technology is known as one of the innovations that can reduce the use of Portland cement in the building industry. It can also give several advantages, such as reducing CO<sub>2</sub> emissions, having a high level of workability, more resistant to chemical attack, and resistant to high temperatures [1-2]. The research of [3-4] attempted to utilize waste material as an alternative for repair material. Study of [5] investigated the effect of alkali solutions on geopolymer properties. The development of geopolymer concrete technology has the potential to increase sustainable infrastructure development to achieve human welfare.

One of the most widely available and utilized aluminosilicate mineral materials is fly ash. Around one billion tons of fly ash waste is produced annually worldwide in coal-fired steam power plants [6]. Most of these by-products are disposed of in nature without treatment, causing environmental damage. Even though in small amounts, fly ash disposal can result in water, soil, and air pollution [7]. It contains several toxic or mutagenic constituents that have a negative impact

on environmental health [8-9]. For the above reasons, there is a growing demand for the utilization of residue of the coal-fired power plant.

In previous work [10-11], the researcher investigated the utilization of low calcium fly ash for alkali-activated (geopolymer) matrices. Researchers developed low calcium fly ash, ground granulated blast furnace slag (GGBFS), and micro-silica as binding materials for alkali-activated mortar [12-13]. There has been no paper discussing the reliability of strength of alkali-activated (geopolymer) mortars by using Acoustic Emission. Therefore, this study applies Acoustic Emission (AE) hits for evaluation of alkali-activated mortar (AAM) reliability under the compressive strength test. This AE test was conducted at Kumamoto University in Japan.

The Acoustic Emission (AE) technique has been applied to many cases for evaluating concrete material. It is one of non-destructive testing (NDT) types, which can be used in materials such as concrete to evaluate damage or fracture occurring not only on the surface but also deep inside the material both qualitatively and quantitatively [14]. AE technique is usually applied during loading, while most other NDT applied before or after

loading of a structure. The purposes of AE tests are to quantify the characteristics of waveforms of AE signals using various methods and to parameterize them, to predict the event, and determine the number and frequency of AE signals [15].

Hit, one of the acoustic emission parameters, is a signal that exceeds the threshold, causing a system channel for data accumulating. It shows an activities of Acoustic Emission with a counted number for a rate [16]. An amplitude, other signals of the acoustic emission parameter, is interpreted by a peak voltage on signal waveform. Magnitudes of Acoustic Emission amplitudes are frequently analyzed in relation to the frequency distribution [17].

In this study, AE test was applied during the compressive load. Acoustic Emission (AE) is used for monitoring the process of material crack propagations to investigate material quality. This study also uses Weibull distribution to analyze Acoustic Emission (AE) hits on AAM and PC mortars under the compressive strength test. This topic has never been published in other research.

## 2. MATERIAL, MIXTURE AND METHOD

### 2.1 Material and Mixture Compositions

Seven-types of alkali-activated mortars and one-type of PC mortar were used for the reliability analysis by acoustic emission. The reliability analysis is performed using Weibull distribution. The compressive test was carried out using cylindrical specimens of 50x100 mm as shown in Fig. 1.

The materials used in this study: PC in accordance with [18] and corresponding to [19]; Japanese fly-ash (F) type II as [20]; ground granulated blast furnace slag (GGBFS) with particle fineness grade 4000-4120 cm<sup>2</sup>/g; and un-densified micro-silica (M) Grade 940 as [21]. Fine aggregate was standardized sand as [22-23]. Each material properties can be seen in Table 1. Material precursor proportions of AAM and PC is shown in Table 2.

AAM mortars are mixtures made from precursor materials (fly ash/GGBFS/micro-silica), mixed with a fixed alkaline solution (Na<sub>2</sub>SiO<sub>3</sub>+NaOH), and standardized sand. All of these materials are then compacted, demolded, cured as previous papers [10-13]. Fixed alkali solution ratio of Na<sub>2</sub>SiO<sub>3</sub> to 8 M NaOH of 1.5 is used. The Na<sub>2</sub>SiO<sub>3</sub> consisted of 17-19% of Na<sub>2</sub>O, 35-38% of SiO<sub>2</sub>, and 46% of H<sub>2</sub>O, by weight. A mixture of PC mortar of 887.8 cc was mixed, compacted, demolded, and cured as [22].

Table 1 Chemical compound and specific gravity ( $\gamma$ ) of materials in weight percentage from XRF test and chemical analysis of standardized sand [13]

Materials	Chemical Compound (% weight)									$\gamma$ (g/cc)
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Na <sub>2</sub> O	H <sub>2</sub> O	K <sub>2</sub> O	MgO	SO <sub>3</sub>	
Fly ash	55.19	25.35	7.57	4.06	-	-	-	-	-	2.33
GGBFS	35	16	0.7	46	-	-	-	-	-	2.89
Micro-silica	93.67	0.83	1.3	0.31	0.4	-	-	-	-	2.22
Standardized Sand	98.4	0.41	0.36	0.16	0.01	<0.2	0.01	-	-	2.64
PC cement	19.74	7.4	2.95	61.9	0.48	-	-	2.87	4.66	3.16
Na <sub>2</sub> SiO <sub>3</sub> gel	36.4	-	-	-	18.5	45.1	-	-	-	1.6
NaOH solution										1.22
Air entraining agent										1

Table 2 Material precursor of mortar proportions [13]

Mortar	Symbols	FA (%)	GGBFS (%)	M (%)
AAM-I	FA <sub>50</sub> S <sub>50</sub>	50	50	0
AAM-II	FA <sub>90</sub> M <sub>10</sub>	90	0	10
AAM-III	FA <sub>40</sub> S <sub>50</sub> M <sub>10</sub>	40	50	10
AAM-IV	FA <sub>47.5</sub> S <sub>47.5</sub> M <sub>5</sub>	47.5	47.5	5
AAM-V	FA <sub>45</sub> S <sub>45</sub> M <sub>10</sub>	45	45	10
AAM-VI	FA <sub>42.5</sub> S <sub>42.5</sub> M <sub>15</sub>	42.5	42.5	15
AAM-VII	FA <sub>40</sub> S <sub>40</sub> M <sub>20</sub>	40	40	20
PC	As reference			

Note: this table illustrates raw (solid) precursor proportions by weight applied in alkali-activated mortars (AAM) with FA = fly ash, S= GGBFS, M= micro-silica and PC = Portland Cement Mortar as control

## 2.2 Experimental Method

AE measurement test was performed during the compressive loading test of cylindrical specimens. Fig. 1 shows the set up for a compressive test using AE. One wide-band UT 1000 AE sensors were attached to the mortar sample's surfaces. AE signals in this work were amplified at 40dB; the threshold level was also set at 40dB. This arrangement is based on the trial method used in the previous experiments, which showed that the amplitude below 40dB is only noise. AE measurement test was conducted using MISTRAS NDT system. Mortar samples were analyzed at zero loads and until it reached its failure point. However, the maximum compressive strength was set to 200 KN, which was equal to 5 kV.

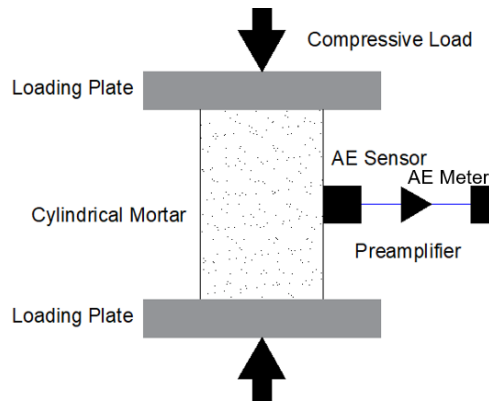


Fig.1 Acoustic emission test set-up on compressive test.

## 3. RESULT AND DISCUSSION

### 3.1 Mechanical Properties

Figure 2 shows the compressive strength of cylindrical mortar specimens at 14 and 28 days. It shows that the compressive strength of all AAM mortar specimens (IV to VII) have higher strength

than that of PC mortar at 14 days. This high early strength is caused by the replacement of more than 40% of fly ash with GGBFS. GBFS will accelerate the reaction at an early age due to the presence of more CaO in the mixture. This result is in accordance with papers [24-25] on the effect of the addition of slag from 10 to 40% on the compressive strength of fly-ash alkali-activated specimens. The more percentage of slag added to alkali-activated fly ash, the higher the compressive strength achieved at an early age. This is in line with the study of [5] which shows that slag enhanced the compressive strength of geopolymer paste due to additional CSH formation.

Figure 2 also indicates that as the micro-silica proportion increases from 5% to 15%, the compressive strength of cylinders' mortars increases only slightly at 28-days. Further, when the micro-silica proportion increase to 20%, the 28-days compressive strength of AAM mortar decreases. On the contrary, PC mortar specimens increase significantly up to 25% for the same period and also it has the highest strength for all mortar specimens at 28 days.

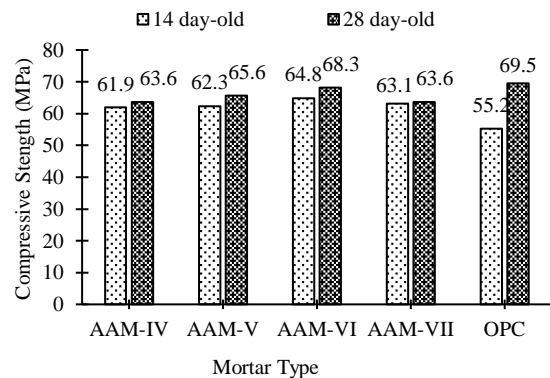


Fig. 2 Compressive strength of cylindrical mortar samples

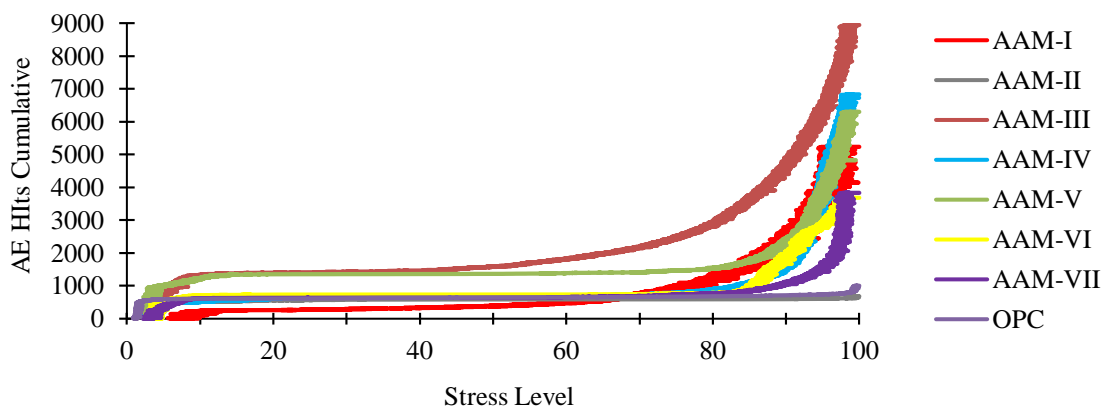


Fig. 3 Acoustic emission hits cumulative vs. stress level of AAM-I to VII and PC mortar

### 3.2 Frequency of Hits and Amplitude of Acoustic Emission under compressive stress load

Initial stress level is recorded when Acoustic Emission generating micro activity during the compressive loading test. Generally, the first period of stress level is associated with the low acoustic emission activities, when the mortar cylinder has just been at the beginning of the compressive test. The continued period is a stage with an increase in Acoustic Emission activities when the compressive load continues to increase on cylindrical specimens. The last period is marked by a significantly increase in Acoustic Emission activities when cylindrical specimens are subjected to a maximum compressive test until failure conditions, such as shown in Figure 3.

Figure 3 shows the relation between the number of AE hits and stress level during compressive loading on a series of alkali-activated mortars (AAM-I to AAM-VII) and one OPC mortar. All the mortars have a low number of AE hits at the beginning of the test. The number of AE hits then increases tremendously as the test reaches the final stress level. Figure 3 indicates that the number of AE hits increase slightly at the beginning of the test until the test reaches about 85% of the final stress for AAM-I and III. The same figure also shows that the number of AE hits increase slightly at the beginning of the test until it reaches about 95% of final stress for AAM II, IV, V, VI, and VII. By contrast, OPC mortar has the number of AE hits increase very low at the beginning of the test until it reaches about 98% of final stress.

The compressive load propagates inside the mortar specimen. Compressive stresses are generated until final stress. AE activities were monitored until 100% of the stress level against the strength. Micro-cracks are generated at a low level of stress. AE activities which occur at stress level in the range from 0 to 85% or 95% are mostly

generated due to the friction of existing micro-cracks. After this stage, AE activities are not only caused by existing micro-cracks, but also by newly nucleated cracks due to the increase of compressive load.

Micro-cracks occur slowly during a lower stress level, followed by the significant increase of AE hits at the final stress level. However, the graph of AAM-II shows that high AE hits occur at the beginning of compressive loading. It was caused by the noise of the environment during the test. This phenomenon was also found at AAM-III to AAM-VII and OPC mortar. Therefore, in general, AE activity of alkali-activated mortars is very similar to that of OPC mortar.

Figure 4 presents the recorded amplitude frequency of hits, where most of the recorded amplitudes in the Acoustic Emission was presented in the data. It shows the frequency of Acoustic Emission amplitude of AAM-I cylinders which then used to determine the intensity of acoustic emission amplitude that occurs during a compressive loading test. The most recorded amplitude is 50dB, followed by 60 or 40 and then 70dB. Therefore, the data were analyzed by Weibull distribution using an amplitude filter greater than 50, 60, 70 and 80dB, because the 40dB was considered as noise in the early measurement.

### 3.3 Weibull Analysis of Amplitude Acoustic Emission

Weibull distribution analysis has the ability to provide a fairly accurate failure analysis with a few sample quantities. The various m values of Weibull distribution analysis from acoustic emission data gives various quality specimens under a compressive loading test. When m parameter from AE generation becomes smaller than 1 means that a high activity occurs at low-level stress, resulting in lower robustness of mortar.

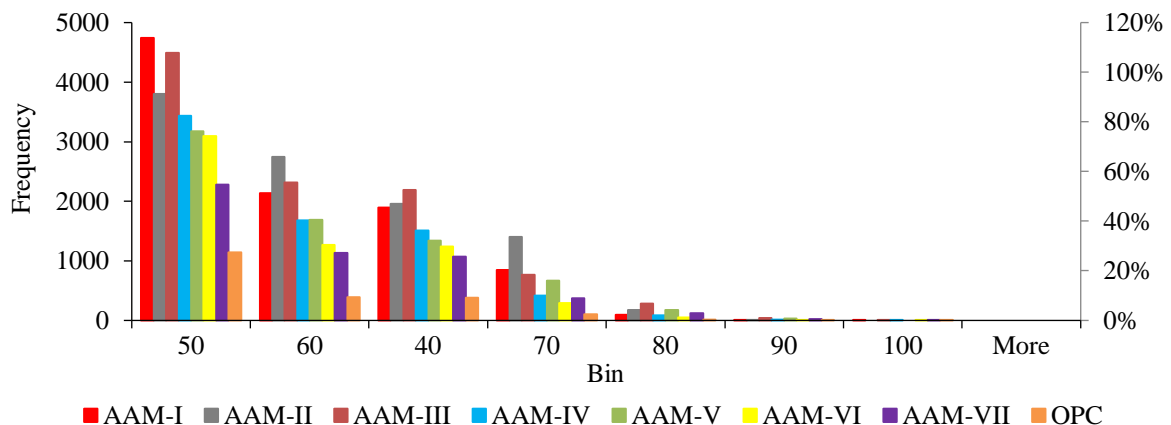


Fig. 4 Histogram Frequency of Hits vs. Amplitude of AAM-I to VII and OPC mortar

The weibull distribution gives an indication that specimen with a lower  $m$  value has a higher probability of failure under compressive strength or lower reliability.

Figure 5 shows an example of Weibull modulus ( $m$ ) values of mortar cylinders. The graph shows gradient ( $m$ ) values of three samples of AAM-I are almost similar when data analysis uses amplitude filter greater than 50, 60, and 70dB, as shown in Fig 5, (a), (b) and (c). Graph (a) with amplitude filter greater than 50dB shows that all cylinders have similar of  $m$  values of 0.94, 1.27, and 1.10, for cylinder 1, 2, and 3, respectively. This indicated that all three cylinders have a similar probability of failure when subjected to a compressive loading test. Figure 5 (b) and (c) also have the same trend as Figure 5 (a). By contrast, Figure 5 (d) shows  $m$  values lower than 1.0 for the three-cylinder, when data analyzing conducted using amplitude filter

greater than 80dB. The graph exhibits lower  $m$  values than that graph that use amplitude filter greater than 50, 60, or 70dB. Cylinders with lower  $m$  values imply that they have a higher probability of failure under compressive strength.

The acoustic emission data under compressive strength for AAM-I are analyzed by Weibull analysis with three-kind filter amplitude (greater than 50, 60, 70dB). It reveals that all AAM-I cylinders have the same quality as indicated by the similarity in  $m$  values. Meanwhile, the amplitude of the filter greater than 80dB shows a slight difference in the value of  $m$ . It is caused by small numbers of data of amplitude greater than 80dB. The  $m$  values of three-AAM-I cylinders are similar because they were made from the same compositions, and therefore have the same quality and will show the same behavior at failure.

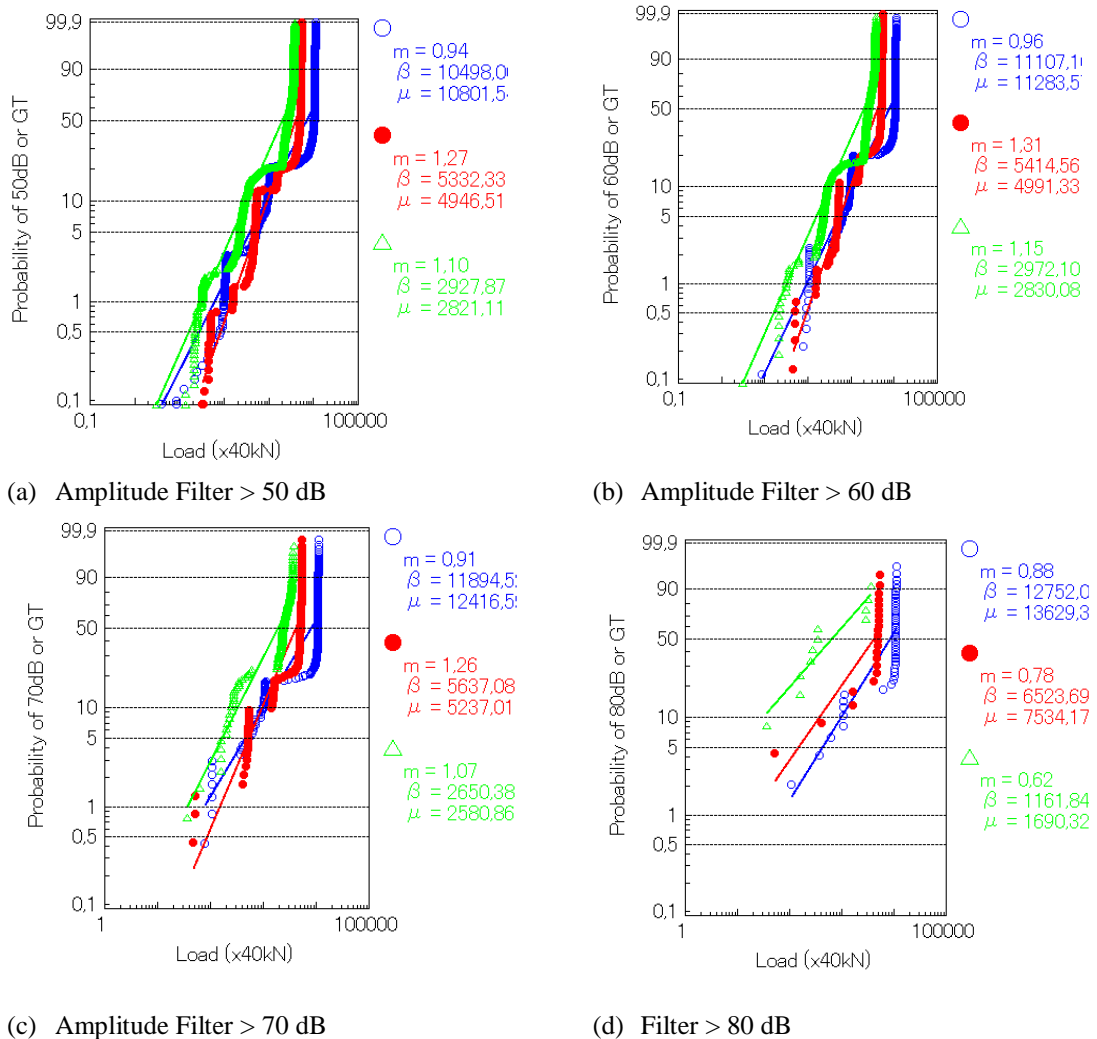


Fig. 5 An example of the probability of failure under compressive loading of mortar with amplitude filter is greater than 50, 60, 70 and 80dB

From these results, it is known that the use of amplitude filter greater than 50, 60, and 70 dB are more accurate for Weibull analysis than that of 80dB. The m values obtained shows that the AAM-I type is in good condition/healthy and robust.

This work also evaluates Weibull modulus (m) on alkali-activated mortar series AAM-II. The m values of the three-AAM-II cylinders are 0.96, 1.2, and 1.1, respectively, when data analysis uses an amplitude filter greater than 50dB. The use of filter greater than 60dB of AAM-II results in m values of 3 specimens of 1.06, 1.33, and 1.23, respectively. When filter greater than 70dB is used the m values obtained is 1.17, 1.22, and 1.19, respectively. However, the use of filter greater than 80 dB gives a significant different three-m value of 0.48, 10.74, and 21.10, respectively, even when used in the same quality specimens. This fact shows that filter greater than 80dB is not suitable to be used for analyzing the data. The Weibull modulus results show that the AAM-II type is in good condition/healthy and robust.

AAM-III cylinders have gradient (m) values of three samples are very similar, when data analysis used amplitude filter greater than 50, 60, 70, and 80dB. The m values show that the AAM-III type is in good condition/healthy and robust.

Gradient (m) values of three samples of AAM-IV and V are also very similar, when data analysis used amplitude filter greater than that of 60 and 70 dB. However, a lower m value was obtained with a filter greater than 50 dB and 80dB. This shows that filter greater than 60 and 70dB is more accurate than that of 50 and 80 dB. The m values show that the AAM-IV type is also in good condition.

The further test shows that m values of three samples of AAM-VI are slightly different. However, only the graph from filter greater than 60dB is suitable for data analysis. The m values of AAM-VI are slightly higher than the other AAM types with filter greater than 60 dB. The m values show that the AAM-VI type is in better condition; more robust, more sound than the other AAM types.

Finally, the test shows that the average m values of AAM-VII are lower than 1. Compared to other AAM types with filter greater than 60dB, AAM-VII has a slightly lower m value. It can be inferred that AAM-VII is not in a better condition than other AAM types. This means that a high activity occurs at a low level of stress, resulting in lower robustness of mortar. It has a higher probability of failure under compressive strength than the other AAM specimens.

For comparison, the AE test is also performed on OPC (Ordinary Portland Cement) mortar. The test shows that the average m values are lower than 1.0. Compared to all AAM types with all filter, OPC mortar has a lower m value. It can be inferred that OPC mortar is not in better condition than that of all

AAM types. A high activity occurs at low-level stress, resulting in lower robustness of OPC mortar. Therefore, OPC mortar has a higher probability of failure under compressive strength tests when compared with all AAM.

#### **4. CONCLUSIONS**

Based on the overall work, the following conclusions could be drawn:

- a. The suitable amplitude filter to examine the reliability of mortar under compressive stress using acoustic emission is filter greater than 60dB.
- b. The various Weibull gradient values indicate that alkali-activated mortars made from fly ash/GGBFS/micro-silica have better condition against compressive loading when compared with Portland cement mortar.
- c. Alkali-activated mortars which contain 40% fly ash/ 40% GGBFS/ 20% micro-silica has lower quality than those of other AAM which contains lower micro-silica.
- d. Alkali-activated mortars which contain 42.5% fly ash/ 42.5% GGBFS/ 15% micro-silica has higher quality than those of other mortars. This mixture is recommended for mix design. It provides high compressive strength and quality.
- e. Portland cement mortar has the highest probability of failure than the other alkali-activated mortars. This shows that the Portland cement mortar has the lowest quality compared to other mortars.

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