Modern Applied Science; Vol. 8, No. 6; 2014
ISSN 1913-1844 E-ISSN 1913-1852
Published by Canadian Center of Science and Education

Reliability Analysis of Clay Ceramics Incorporating Industrial Solid By-Products

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Received: August 26, 2014 Accepted: September 8, 2014 Online Published: November 8, 2014

doi:10.5539/mas.v8n6p219 URL: http://dx.doi.org/10.5539/mas.v8n6p219

Abstract

The Weibull statistics are extensively used in the last years to estimate the reliability of fracture data of brittle construction materials such as ceramics, which tend to present a large scatter in mechanical strength. In the present research, clay-based building ceramics incorporating industrial solid by-products were fabricated employing a pilot-plant simulation of industrial brick manufacturing and their flexural strength was evaluated by three-point bend testing, before and after subjecting the bricks to 25-cycle frost resistance testing. Then, the modulus of rupture was calculated and analyzed using Weibull distribution. Industrial powders including different fly ash samples and steel making dust were utilized as substitute secondary resources, mixed with standard brick clays and water, and extruded into rectangular brick specimens, which were sintered in a controlled furnace. Weibull plots of the results allow the prediction of the stress, at which the probability of survival of the ceramic bodies is kept to a certain value. The relative clay/by-product mixture composition, sintering temperature and residual porosity are proved to affect the brick bending strength and reliability. Porosity depends on the specific pore-forming agent used as well as on the sintering temperature. In conclusion, Weibull analysis appears to be of particular importance to assess the quality of ceramics incorporating industrial solid by-products.

Keywords: reliability analysis, Weibull statistics, bending strength, clay ceramics, industrial solid by-products

1. Introduction

Ceramics have been widely used as construction materials for their excellent properties. Their drawback, however, is the inherent brittleness. Moreover, they not only break easily but also tend to present a large and unpredictable variation in mechanical strength. Nevertheless, strength reliability is one of the critical factors restricting wider use of brittle materials in various structural applications. This leads to the necessity of employing statistical analysis in view of their utilization in design and manufacturing. Actually, analysis of fracture data in ceramics can be made by applying probabilistic theories. Among other distributions, the Weibull statistics (Weibull, 1951) has a strong theoretical basis and is used in the last years to estimate both the distribution and reliability of fracture data of brittle materials. Although there is not sufficient evidence that the Weibull distribution, being valid only for a special set of material conditions and in a limited parameter space, should always be preferred to other distributions, this theory is up to now the backbone in the design of brittle components, as it is usually reported to most accurately describe the strength scatter, when compared to different other theoretical statistical distributions (Danzer et al., 2007; Gorjan & Ambrožič, 2012; Härtelt et al., 2012; Lu et al., 2002).

From the literature review of theoretical foundations and applications of Weibull analysis (Aarseth & Prestlokken, 2003; Askeland et al., 2011; Basu et al., 2009; Du et al. 2012; Fu et al., 2014; Loidl & Peterlik, 2010; Oikonomou et al., 2007), it results that Weibull statistics is based on the weakest link hypothesis and assumes that fracture in ceramics begins from many micro-flaws behaving like micro-cracks, sparsely distributed throughout a material, which have random positions, shapes, sizes and orientations. It is further assumed that such cracks do not interact, this meaning that their separation is large enough for their stress fields not to overlap. Variation in sizes of pre-existing flaws/cracks in the bulk of the material results in high scatter in strength, as such flaws/cracks prevent a homogeneous distribution of stresses in the material, hence rupture is caused by stress concentrations at the tip of the cracks.

In order to evaluate flexural strength of ceramics using Weibull analysis, a simple uniaxial three-point or four-point bending test on rectangular specimens is usually preferred, as the bending strength can be easily calculated from the measurement geometry and the breaking load. Another consideration is the increased number of test specimens that might be needed to characterize an entire Weibull strength data distribution rather than simply estimate a mean strength value. Actually, the lower limit of test specimen number should be more than 20 specimens to decrease the scatter of shape parameter, while approximately 30 test specimens are considered to provide adequate Weibull strength distribution parameters, as a general rule-of-thumb in the absence of specific requirements. Furthermore, meticulous specimen preparation and diligence in test implementation are particularly important when using Weibull statistics: ceramic strength data must be essentially free of any experimental error, which should not contribute to a valid data set for Weibull analysis (Ambrožič et al., 2008; McNamara et al., 2014; Quinn & Quinn, 2010; Xu et al., 2001).

On the other hand, secondary useful resources are currently under consideration as substitute raw materials or as admixtures into clays for manufacturing building ceramics as well as geopolymers and composites (Álvarez-Ayuso et al., 2008; Badanoiu & Voicu, 2011; Karayannis et al., 2013; Karayannis et al., 2012; Komnitsas et al., 2009; Konin & Kouadio, 2012; Papanicolaou et al., 2011; Zhang, 2013).

In the present work:

- Rectangular ceramic specimens from clayey raw material mixtures incorporating industrial solid by-products were extruded and fired and the flexural strength of the consolidated specimens was evaluated by three-point bending, before and after subjecting them to 25-cycle frost resistance testing. It should be noted that frost resistance is directly associated with microstructure and mechanical properties (including bending strength) of the final product.

- Then, the modulus of rupture was modeled using Weibull distribution analysis, and the Weibull parameters were estimated and studied in relation to the % admixture content and the firing temperature, in order to assess the quality of the ceramics produced, while also making an appropriate use of material and energy resources.

- The beneficial utilization of industrial powders in the manufacturing of value-added construction materials can contribute to both production cost reduction and environmental management and protection.

2. Raw Materials

Industrial powders containing several valuable oxides were utilized as the substitute raw materials and incorporated in clays typically used by the ceramic industry. Specifically:

Fly ash derived from conventional burner (FA): Fly ashes are annually produced in huge amounts from lignite/coal combustion in power units (Adamidou et al., 2007; Karakasi & Moutsatsou, 2010; Karayannis & Moutsatsou, 2012). Hence, the management of these by-products is nowadays of great environmental concern. The fly ash sample used was derived from conventional lignite burning in thermal power station of Western Macedonia Region - Greece. This ash is highly calcareous (approx. 25 wt.% CaO content) and can be classified as Class-C (ASTM). It also comprises noticeable amounts of sulphur (7 wt.% SO_3 content).

Fly ash from circulating fluidized bed combustion (CFB): The quantities of fly ash globally produced from fluidized bed-coal combustion for power generation are also steadily growing (Mei et al., 2014), as this technology – and particularly its advanced part, circulating fluidized bed (CFB) combustion – continuously gains ground for environmentally-friendlier power production. Due to the intrinsic characteristics of CFB-technology, the fly ashes formed can be different from those generated in the conventional processes. The CFB-fly ash used in the present research is clearly siliceous (approx. 40 wt.% SiO_2 content) with also a significant CaO percentage. Similarly to other ashes derived from CFB-combustors, it also contains considerable amounts of sulphur.

Steel-making dust (EAFD): The use of electric arc furnace technology in the steel-making industry has been increasing considerably over the last decades resulting in the production of significant quantities of solid residues

(Karayannis et al., 2011). Particularly in electric arc furnace dust (EAFD), a solid waste from gas treatment, which was also used in the current study, main oxides found are FeO and ZnO (over 50 wt.% of the dust).

3. Experimental

3.1 Specimen Fabrication

Clay-based brick-shaped specimens were fabricated employing a laboratory pilot-plant simulation (see flowchart in Figure 1) of industrial brick manufacturing procedures (Domopoulou et al., 2014; Spiliotis et al., 2014; Spiliotis et al., 2013): the powdery by-products were mixed with the clayey raw materials and subsequently kneaded with water and shaped into rectangular brick specimens by plastic extrusion. The so-produced green specimens were dried and then fired for sintering and consolidation in a temperature controlled furnace.

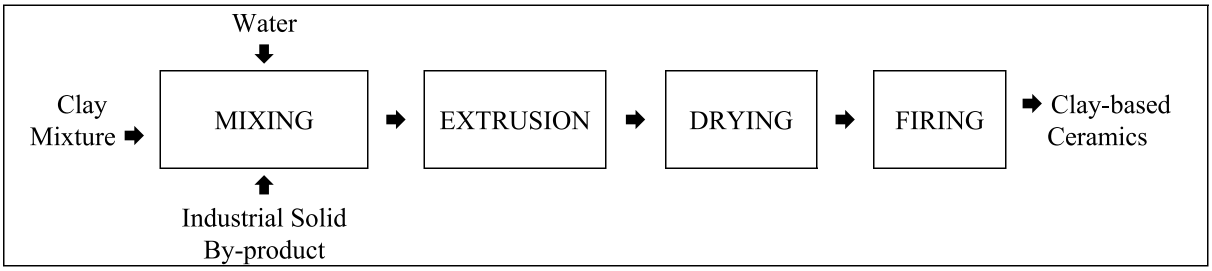

Figure 1. Flowchart of the method employed for fabrication of clay-based ceramics

3.2 Specimen Testing

Three-point Bending Strength: The flexural strength of the fired specimens was evaluated by 3-point bending, before and after subjecting them to 25-cycle frost resistance testing. Since a sufficient number of measurements are required in order to obtain a certain accuracy and the measured values be treated statistically taking into account the expected Weibull distribution, 30 specimens were tested for each specific experimental set to interpret the results correctly. The Modulus of Rupture (M.O.R.) in MPa upon 3-point bending testing of the sintered specimens was calculated from the following equation:

$$M.O.R. = (3PL)/(2bw^2) \quad (1)$$

where: P = force (MN)

L = the opening width (0.036 m)

b = width of specimen (m)

w = thickness of specimen (m)

Then, the M.O.R. results were analyzed using Weibull statistics. The fit of data to a Weibull distribution can be visually assessed using a Weibull plot of the empirical cumulative distribution function of data on special axes, a graphical technique for determining if a data set comes from a population that would be fit by a Weibull distribution. The Weibull plot is formed as follows:

Vertical axis: Weibull cumulative probability expressed as a percentage

Horizontal axis: ordered failure times (in a log10 scale)

The vertical scale is $\ln(-\ln(1-P_f))$, where $P_f=(i-0.3)/(n+0.4)$ and i is the rank of the observation

Failure probability: $P_f=1-\exp(-(\sigma/\sigma_o)m)$

Survival probability: $P_s= \exp(-(\sigma/\sigma_o)m)$

m: the Weibull modulus (shape parameter)

σ_o: characteristic strength

Water Absorption Capacity: The water absorption capacity of bricks is directly related to their open porosity that in turn influences mechanical properties. In the present research, water absorptivity was determined by specimen immersion water (25-30^{o}C) for 24 hours. Water absorption (WA %) was calculated from the following equation:

$$WA\ (\%) = 100\ (W_{wet}–W_{dry})/W_{dry} \quad (2)$$

where: W_{wet} = weight of saturated with water samples (g)

W_{dry} = weight of dry samples (g)

Frost Resistance: One of the criteria of ceramics quality is their low temperature stability. The frost resistance of the brick specimens produced was examined by freezing/thawing procedure (25 cycles) with the following steps:

a. water immersion at room temperature for 72 hours

b. cooling to 0°C in about 110 min

c. stay at 0°C for 1 h

d. further cooling down to -15°C and stay at that temperature for 1 hour

e. quick heating to room temperature and stay for 1 hour at 25°C

4. Results and Discussion

4.1 Fly Ash from Conventional Burner (FA)

Figure 2 depicts the stress survival probability (Ps) distribution (reliability versus strength diagram) and Figure 3 the variation of Weibull parameters, namely a) the Weibull modulus (m), b) the characteristic strength (σ_o), c) the mean value (σ_μ), d) the strength corresponding to 90% Ps (σ_{90}) and e) the water absorption capacity (WA %), for clay-based brick specimens with different % FA content, all sintered at 1050°C. It should be specified that the Weibull modulus (m) describes the scatter of the data (small values of m would denote large scatter in strength).

In Figure 2, almost all the Ps distribution curves present similar slopes. A better bending strength reliability results for brick specimens containing FA in the range of 0-6 wt.%, while it deteriorates for higher FA percentages. Therefore, the optimum % FA content should be near 6 wt.%.

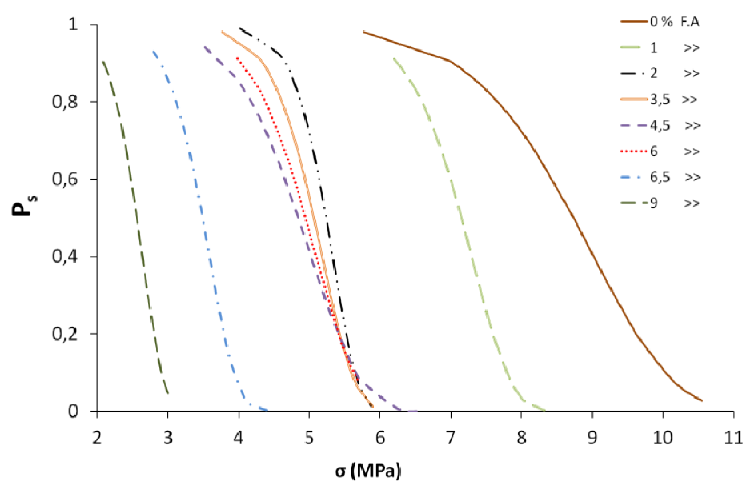

Figure 2. Influence of % FA addition on bending strength reliability of clay-based ceramics, P_s=f(σ) (T_{sint}=1050°C)

The results in Figure 3 show that m values for all specimen sets are near or even higher than 10, a value corresponding to engineering ceramics with relative low data scattering. It should be noticed here that traditional ceramics typically have m values about 5 or lower, and thereby strength data remain reliable even for specimens incorporating up to 9 wt.% FA. However, for modern technical ceramics m values between 10 and 20 and even higher have been reported (Askeland et al., 2011; Bermejo et al., 2012; Danzer et al., 2008; Kübler & Gauckler, 2010).

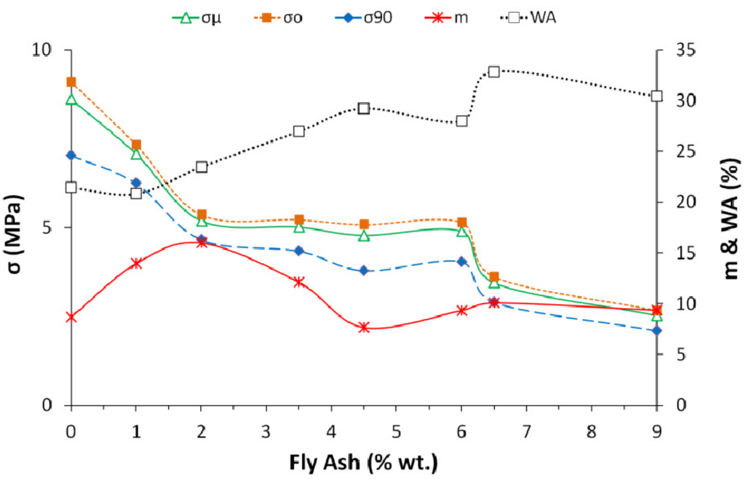

Figure 3. Weibull modulus (m), stress values (σ_μ, σ_o, σ_{90}) and water absorption (WA%) of clay-based ceramics as a function of their % FA content (T_{sint}=1050^{o}C)

The Weibull characteristic strength (σ_o) and the mean stress value (σ_μ) are similar, while σ_{90} is lower, and the higher differences between them appear for the specimen sets exhibiting the lower m values. All stress parameters generally follow reverse route with the brick water absorption capacity (WA %). Actually, taking into account that generally higher WA % reveals the presence of more and larger flaws in brick body, a degradation in strength reliability, thus a reduction in σ_o and σ_{90}, is normally attended when WA % increases and vice versa.

Figure 4 shows the effect of 25-cycle frost resistance testing on the Weibull parameters for clay-based brick specimens with different % FA content, all sintered at 1050^{o}C.

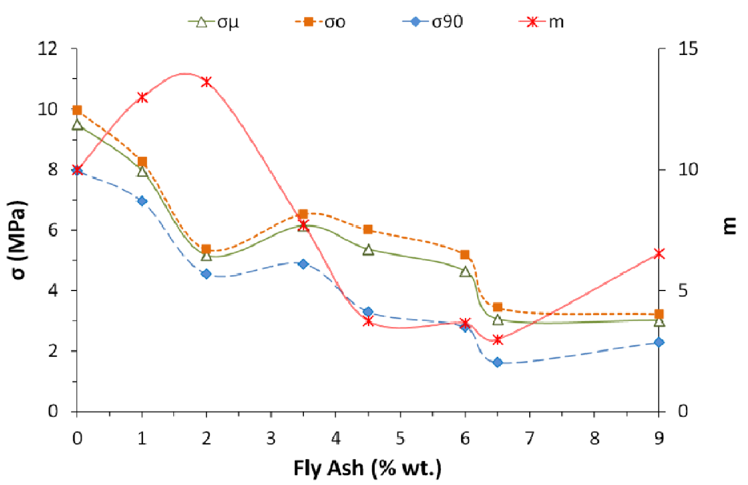

Figure 4. Weibull modulus (m) and stress values (σ_μ, σ_o, σ_{90}) of clay-based ceramics as a function of their % FA content (T_{sint}=1050^{o}C), after 25-cycle frost resistance testing

According to the results presented in Figure 4, for a FA content up to 3.5 wt.%, m values remain at high levels, while further FA addition in the clayey raw materials leads to significantly lower m (<5 for FA content between 4.5 and 6.5 wt.%), thus more scattered strength data. Unexpectedly however, for even higher % FA content, up to 9 wt.%, m tends to increase implying a decrease in data scattering. To understand this behaviour, a more intense presence of glassy phase, inherent in FA, in the bricks with the higher FA percentages should be taken into consideration, as such glass-containing systems are reported to show better frost resistance characteristics in comparison with the kaolinite system (Molnar et al., 2003). The obtained results might also be attributed to calcium-silica hydrates formation during cycling treatment (Sidjanin et al., 2007), enhanced by the strongly calcareous character of the FA used, and causing a different behaviour of this kind of systems.

Moreover, comparing m values from Figures 2 and 4 for specimen bending strength before and after frost resistance testing respectively, it is obvious that freeze-thaw cycles deteriorate strength scattering by a mechanism which probably enhances the already existing flaws and possibly creates new ones.

In Figure 5, the effect of sintering temperature on Weibull parameters as well as on water absorption capacity of brick specimens with the optimum FA content (6 wt.%), before and after subjecting them to 25-cycle freeze-thaw testing, is presented.

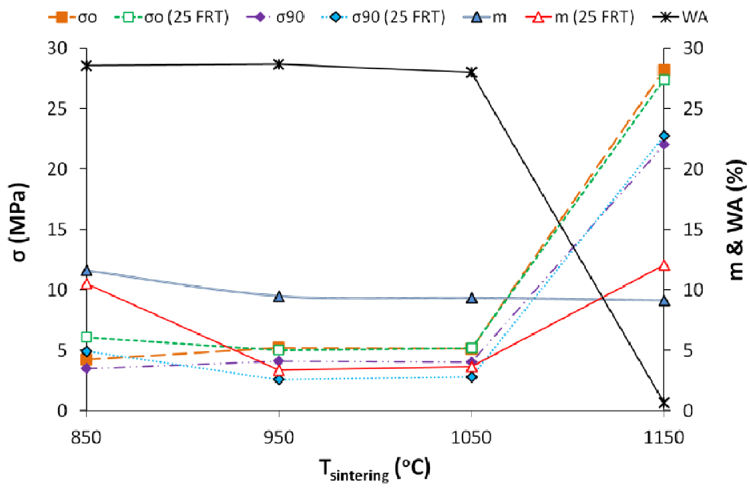

Figure 5. Influence of sintering temperature on Weibull modulus (m), stress values (σ_{μ}, σ_o, σ_{90}) & water absorption (WA%) of 6% FA content ceramics, before & after subjecting them to 25-cycle freeze-thaw testing (25FRT)

Figure 5 shows that, before the frost resistance testing, Weibull modulus (m) remains practically constant (~10), thus indicating a constant and high reliability, independent of the firing temperature. After the implementation of 25-cycle freezing-thawing, m decreases rapidly, when the sintering temperature is increased from 850°C to 950°C. However, at 1150°C firing, elevated values of w, σ_{μ}, σ_o, σ_{90} are obtained, which can be attributed to a practically eliminated open porosity, as indicated by the sharp increase in specimen WA (%) capacity respectively.

4.2 Fly Ash from Circulating Fluidized Bed Combustor (CFB)

Figure 6 illustrates the effect of %CFB addition on Weibull parameters as well as on water absorption capacity of brick specimens sintered at 1050°C, before and after subjecting them to 25-cycle frost resistance testing.

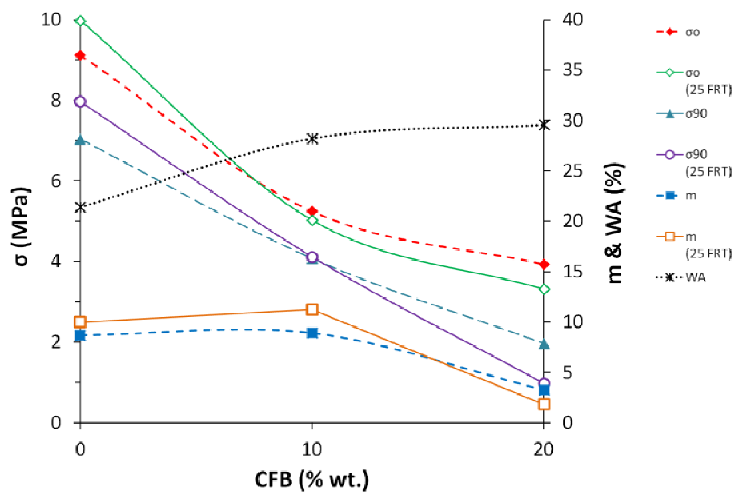

Figure 6. Influence of %CFB addition on Weibull modulus (m), stress values (σ_{μ}, σ_o, σ_{90}) & water absorption (WA%) of clay-based ceramics (T_{sint}=1050 °C), before and after 25-cycle freeze-thaw testing (25FRT)

The results show that the addition of 10 wt.% CFB in clay mixtures does not affect the Weibull modulus (m) of the bricks produced, even after freezing-thawing test. However, when 20 wt.% CFB is embodied in the mixture, clearly more scattering stress data are recorded (m<3.5), a finding that could probably be associated with an increase in open porosity indicated by a noticeable increase in brick water absorptivity observed respectively.

4.3 Steel-Making Dust (EAFD)

In Figure 7, the influence of wt.% EAFD content on Weibull parameters as well as on water absorption capacity of brick specimens produced at 1050°C is presented. According to the results, all Weibull parameters examined are practically unaffected by EAFD addition up to 15 wt.%. WA (%) of the bricks is also not particularly influenced by the incorporation of this specific powdery industrial byproduct, thus indicating a constant open porosity. Hence, EAFD, when added in such proportions, does not act as a pore forming agent in the ceramic body. Concluding, all brick sets of this series provide relatively reliable strength data (m values around 10).

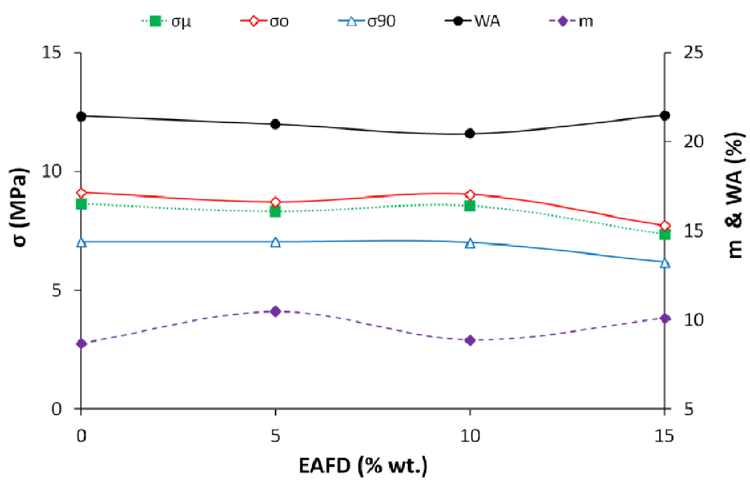

Figure 7. Influence of %EAFD addition on Weibull modulus (m), stress values (σ_{μ}, σ_o, σ_{90}) and water absorption (WA%) of clay-based ceramics (T_{sint}=1050°C)

The effect of firing temperature on Weibull parameters as well as on water absorption capacity of brick specimens containing 10 wt.% EAFD is shown in Figure 8. It is evident from this Figure that the bricks so-produced exhibit similar Weibull parameters and WA %, when the sintering temperature is increased from 850°C to 950°C, while further increase in firing temperature results in more scattering bending strength data.

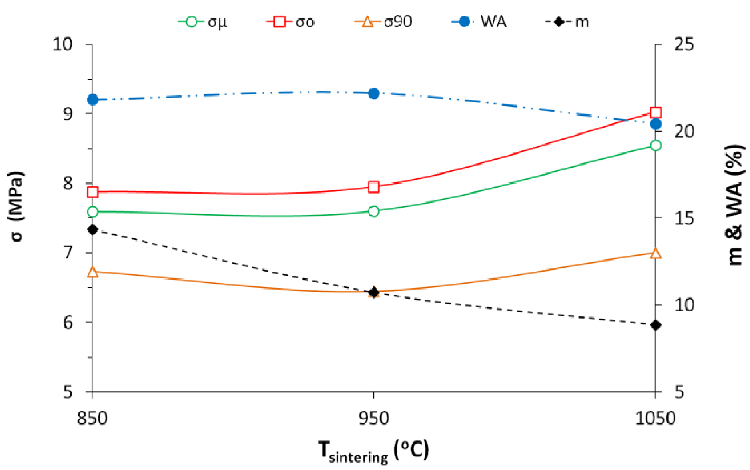

Figure 8. Influence of sintering temperature on Weibull modulus (m), stress values (σ_{μ}, σ_o, σ_{90}) & water absorption (WA%) of 10% EAFD content ceramics

4. Conclusions

Weibull analysis of the three-point bending strength experimental results of clay-based bricks incorporating industrial solid by-products, before and after subjecting them to frost resistance testing, allows the prediction of the stress, at which the probability of survival is kept to a certain value.

Reliable strength results are generally achieved by adding each time 0-10 wt. % either fly ash derived from power station conventional burners, either fly ash from circulating fluidized bed combustion or steel-making dust.

Several parameters, including clay/by-product mixture composition, firing temperature, flaws in the ceramic body and porosity affect the flexural strength and reliability of bricks containing industrial powders. Particularly, open porosity variation depends on the admixture (pore-forming agent) used as well as on the sintering temperature.

In conclusion, Weibull analysis is important to assess the final product quality of building ceramics, while also making an appropriate use of material and energy resources.

Acknowledgments

This research has been co-financed by the European Union (European Social Fund - ESF) and Greek national funds through the Operational Program "Education and Lifelong Learning" of the National Strategic Reference Framework (NSRF) – Research Funding Program: **ARCHIMEDES III**. Investing in knowledge society through the European Social Fund.

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