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Optimization of microstructure and insulation characteristics of basalt-based fibers

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Abstract

Batch compositions of basalt and dolomite were optimized to produce high-quality fibers and wool boards applying Cupola technology. The raw batches, cinders, single fibers and fabricated wool boards have been characterized using XRD, XRF, SEM-EDAX, μ -X-ray CT, Lambda meter, mechanical and acoustic testing procedures according to ASTM and EN standards.

The CaO/MgO ratio, acidity, basicity and melting moduli of the basaltic cinders are mainly related to the basalt low content of Mg-bearing minerals (19.15wt.%). The single fibers diameter (8.60 μ m), non-fibrous shots (13.00wt.%) and Al₂O₃ (14.08wt.%) contents together with the quasi-horizontal macrostructure ($S_{L-C}=0.60$) improve the wool boards compressive (14.29kPa) and tensile (3.00kPa) strength as well as point load (140.00N) properties.

The single fibers microchemistry shows their enrichment with thermal conductivity ceasing oxides ($\Sigma=65.02$ wt.%) which together with the average porosity (92.30%) enhance the thermal (32.43mW/mK and 6.17m²K/W) and acoustic (1.00 α s and 1.05 NRC) wool board insulations.

Keywords: Basalt, fiber, wool, Cupola, insulation.

1. Introduction

The world needs effective thermal insulation of buildings for pollution control and energy savings. Optimum thermal, fire and acoustic insulations are achieved by using the basaltic/rock wool board fibers [1-4]. Commercially, there are two types of wool fibers, the short and continuous ones. The short fibers are those known as rock wool that are used in the formulation of insulation boards and blankets whereas the continuous fibers are mainly involved in the high-performance composites and reinforcement applications due to their better tensile strength [5-8].

The basalt fibers have superior physico-mechanical and chemical characteristics in addition to their cost-effective production when compared with the carbon and silicon carbide fibers [5]. They have high thermal stability, vibration resistance, durability, heat and sound insulations [5, 9-11]. In addition, the basalt fibers are stable in acidic and basic media, non-combustible, explosion-proof and have non-toxic reactions with air or water [5,10,12,13].

The favorable properties of the basalt fibers call for their formulation into insulating wool boards. These boards would be the only fire retardant standing protective shield remaining on buildings steel structures after firing time of 56

minutes with a maximum temperature of 750°C with minimum fire growth, heat release and smoking emission indices [2,13-14].

Basalts as natural rocks can be used for manufacturing of basalt fibers by two industrial approaches, these are: a) the die-blowing/duplex technology and b) the Cupola technology. Basalts are the sole raw material to produce rock fibers by the duplex method where no melting agents are added to the raw basalt [7,15-16]. However, basalts should be mixed with limestones, dolomites, slags - as calcium and magnesium additives - to produce rock fibers by the Cupola furnace technology [15-18]. The Cupola furnace is made of steel vertical cylinder that fed at the top with fuel (coke), basalt and dolomite for fiber production. The air is inserted into the Cupola furnace across tuyeres located close to the bottom of the furnace. As long the coke is burnt the furnace charge drops and melts, producing a continuous flow of basaltic molten lava that comes out from the Cupola siphon exit and fiberized [5,8-9,15]. In duplex technology, the diameter of the single fibers would range from 1.00 to 5.00µm. These fibers are generally more brittle and expensive when compared with those produced by the Cupola [6-7,16,19]. The latter technology produces fibers between 6.00 and 10.00µm diameter and is commonly used in constructional, industrial and agricultural sectors to save energy and significantly reduce greenhouse gas emissions [20-21].

The CaO/MgO ratio, acidity, basicity and melting capability moduli of basalt and the Cupola post-melting basaltic cinders control the characteristics of the single fiber and wool board end products. The fluidity of the basaltic cinder would depend on the Ca/Mg ratio of the basalt. The fluidity of the cinder would in turn impact the extended fiberization temperature range and consequently the crystallization of minute crystals within and among the single fibers [5,10,12].

The operating temperature range of the cinder fiberization is also affected by the ratio of the cinder viscosity or acidic modulus to its surface tension. The higher the viscosity of the cinder, the higher its acidity modulus and the less is the cinder surface tension, i.e., viscous non-sticky cinder. Such cinder of high viscosity to surface tension ratio has stable fiberization with optimum pull rate [5,10,12].

All the calculated moduli are mainly affected by the mineral content of the basalt which should contain a low amount of high melting point minerals, i.e., olivine and magnetite [15]. The existence of such minerals in the basalt would require its melting at elevated temperatures. The high-olivine basalt could not be completely melted during processing where remnants of high melting point minerals such as olivine and pyroxene would continue to exist in the cinder.

Consequently, these high-temperature phases would remain as relics among the single fibers degrading the quality of the formulated wool boards. On the contrary, the low-olivine basaltic cinder would be devoid of high-temperature melting point minerals and vitrify during fiberization without crystallization resulting in fibers with optimum characteristics.

On the other hand, the crystallization of any phase from the basaltic cinder during fiberization is triggered by the cinder concentration of Fe, Ti, Mg and Ca oxides [5,10-12]. The Fe and Ti would act as crystallization centers along which linear growth rate of crystals is achieved. The crystallization of phases would reduce the fiberization operating temperature range and degrade the fibers insulating and mechanical characteristics [5,9,11].

The present work concerns the evaluation of 15 quarried basalts processed industrially by Cupola industrial furnace to produce rock wool boards. It aims at studying the effect of the raw basalt mineralogy, and consequently chemistry, on the processing moduli of the basaltic cinders. In addition, this work looks for the relations among the raw basalt properties to the produced single fibers and the wool boards physico-mechanical, macro- and microstructure, thermal, acoustic and fire insulation specifications according to EN and ASTM standards.

2. Materials and methods

2.1. Materials

The basalt samples were collected from 15 quarries at Abu-Zaabal, El-Fayoum and Bahariya areas, whereas the dolomite sample was collected from Gabal Ataqa, Egypt (Figs. 1a, b). After characterization, the basalt quarry samples were mixed to represent three technological samples nominated as AZ, FA and BA for Abu-Zaabal, El-Fayoum and Bahariya, respectively. Each technological sample was mixed with dolomite into a raw batch fed separately to the Cupola furnace for further processing and production of wool fibers (Fig. 1c). The dolomite addition facilitates the batch melting by decreasing the energy consumption and cinder viscosity with consequent increase of cinder flowability and productivity.

The batch is melted into cinder inside the Cupola furnace at 1500°C. The cinder then passes out to the Cupola siphon exit (Figs. 1d, f, Video 1) to be fiberized into billions of single fibers through four rapid rotating fabrication discs by centrifugal force (Figs. 1g, h, Video 1). The single fibers are sprayed with a thermosetting phenol formaldehyde binding agent through binder distributors (Fig. 1g). Once the fibers are extracted from the fiberizing machines, they are gathered on a collecting drum sheet (Fig. 2a) then conveyed to the pendulum which moves laterally to distribute the single fibers into the mattress (Figs. 2b, c). The latter is conveyed to a crimping machine to be pressed into loose fiber

boards (Fig. 2d). The compression applied by the crimping machine would force the single fibers to have different orientations.

The loose fiber boards are then fed to the curing oven ($\leq 280^{\circ}\text{C}$) to initiate the polymerization reactions of the phenol formaldehyde resin binder (Fig. 2e) and to form wool boards of different densities. The boards are perfectly shaped by longitudinal and transverse cuttings based on the required dimensions (Fig. 2f) then moved to the stacking stage (Fig. 2g) and conveyed to the final packing which is a standard for every manufacturer (Fig. 2h). The whole industrial fiberization process starting with the raw material and ending with the final packing is shown in Video (2).

2.2. Methods

Representative 500g of the raw basalt and dolomite quarry samples were ground separately in an agate ball mill, sieved to pass a $63\mu\text{m}$ sieve and dried overnight at 110°C . The raw basalts, cinders and wool board samples were characterized for their phase and chemical composition using XRD (PANalytical X'Pert Pro) and XRF (Bruker SRS3000 with WDX detecting system). The chemical analyses of the basalt and cinder samples are processed to calculate the CaO/MgO ratio, acidity (M_a), basicity (K_z) as well as melting capability (M_k) moduli according to the following equations:

$$\text{Equation (1): } M_a = m\text{SiO}_2 + m\text{Al}_2\text{O}_3 / m\text{CaO} + m\text{MgO}$$

where:

M_a : Modulus of acidity,

m : Mass content of oxides (wt.%).

$$\text{Equation (2): } K_z = (100 - (m\text{SiO}_2 + m\text{Al}_2\text{O}_3)) / (m\text{SiO}_2 + m\text{Al}_2\text{O}_3)$$

where:

K_z : the basicity modulus,

m : mass per cent of oxides (wt.%).

$$\text{Equation (3): } M_k = m(\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3 + \text{TiO}_2) / m(\text{CaO} + \text{MgO} + \text{Na}_2\text{O} + \text{K}_2\text{O})$$

where:

M_k : the melting capability modulus,

m : mass per cent of oxides (wt.%).

The macro- and microstructure of the raw basalt and the wool boards were investigated using transmitted light and stereo microscopes in addition to SEM (JSM 6300) equipped with a Bruker XFlash 5010 EDAX. The major macrostructure parameters revealing the orientation of fiber bundles are determined based on equation (4):

$$S = \alpha x / \alpha x'$$

where:

α : Average inclination angles of fibers in a cross section parallel to axis “x”,

α' : Average inclination angles of fibers in a cross-section perpendicular to axis “x”,

S: Illustrates the rate of fiber orientation angles in a flat insulation board specimen regarding axes x, x'.

Where “x” axis is considered the main axis coinciding with fiber direction and lateral movement of the pendulum machine.

Selected wool board samples were examined by X-ray micro-computed tomography (3D- μ XCT) using a X-tek XT H225 (Nikon Metrology). Scans were acquired with a beam energy of 90kV, beam current of 100 μ A, with 2500 projections and two frames per projection at an exposure time of 1s. 3D images were reconstructed with the X-tek CT pro software (Nikon Metrology) at a resolution of 9 μ m. All image processing steps were carried out with the Fiji distribution of ImageJ [22]. A cylindrical region of interest with undisturbed structure was selected and filtered with a non-local means filter [23]. The images were segmented into solid and pore space via Hysteresis thresholding in the 3D Image Suite plugin [24]. The quantitative analysis comprises visible porosity directly determined on the segmented images. Pore connectivity and tortuosity among the boards single fibers were obtained with MorphoLibJ [25]. The

volume fraction of pore diameters and fiber diameters were obtained with the maximum inscribed sphere method implemented in the Local Thickness method. After visual inspection, the diameter threshold to distinguish between fibers and shots was set to 110 μ m. The volume fraction of all larger solid diameters comprises the shot volume density. From the remaining fiber volume, a total fiber length density was derived from the known diameter and volume of each diameter class assuming perfectly cylindrical fibers.

The dimensions of the formulated wool boards had been measured to calculate the board density (ρ) according to EN 822. The different density samples are stored for 6hrs at 23 $\pm$ 2°C and 50 $\pm$ 5% relative humidity before any testing to stabilize their weight according to EN 826.

The compressive strength (σ_m) measured for the wool board samples is calculated according to EN 826. The short- and long-term loads of the wool boards are represented by the point load values which is measured according to EN 12430. In addition, the tensile strength of the wool boards is tested in accordance with EN 1607. All the mechanical tests are conducted using the INSTRON load cell 3300 series of mechanical testing systems supported with BLUHILL universal software with $\pm$ 0.50% of reading down to 1/200 of the

load cell capacity and $\pm 1.00\%$ of reading from 1/200 to 1/500 of the load cell capacity (10kN).

The thermal conductivity and resistance are represented by K- and R-values, respectively. Both values are measured using automated lambda meter EP500e according to EN 12667 and ASTM C177 standards, respectively. The selected wool boards had been tested for their fire resistance characteristics according to EN 13501-1 and ASTM E84 whereas the noise reduction coefficient “NRC” and the sound absorption coefficient “ α_s ” are calculated according to EN ISO 354 in a reverberation room.

3. Results and discussion

3.1. Raw basalt characterization

The chemical analyses of the basalt samples show that silica (48.76 - 50.60), magnesia (5.25 - 10.98), calcium (7.55 - 10.07) and iron (10.70 - 12.96wt.%) oxide contents (Table 1) exhibit 0.74, 1.62, 0.69 and 0.80 standard deviation, respectively. The latter values reveal the high degree of chemical homogeneity due to equal proportion mixing of each area single quarry samples into technological ones nominated as AZ, FA and BA referring to the mix of Abu-

Zaabal (A1 to A5), El-Fayoum (F1 to F5) and Bahariya (B1 to B4) basalt samples, respectively (Table 1).

The XRF analyses are used for the accurate quantitative calculations of the potential minerals applying the CIPW Norm method [26]. The CIPW norm shows that the basalt samples are composed mainly of mineral/crystalline (85.97, 86.75 and 86.51wt.%, Table 2) and glass/amorphous (14.03, 13.25 and 13.49wt.%, Table 2) in the AZ, FA and BA samples, respectively (Table 2). The expected CIPW crystalline phases are mainly plagioclases (Ab+An) (45.46, 46.42 and 45.12wt.%, respectively, Table 2) and the Mg-bearing minerals diopside (Di) and hypersthene (Hy) (22.35, 19.15 and 27.55wt.%, respectively, Table 2) dominating the AZ, FA and BA samples, respectively. No olivine is expected by the CIPW in all samples.

The XRD patterns (Fig. 3) proved that AZ, FA and BA samples are mineralogically composed of anorthite-plagioclase ($\text{Al}_{(1.91)}\text{Ca}_{(0.716)}\text{Mn}_{(0.196)}\text{Na}_{(0.045)}\text{Si}_{(2.089)}\text{O}_8$) (pdf 900-5310) and labradorite-plagioclase ($\text{Al}_{(1.67)}\text{Ca}_{(0.67)}\text{Na}_{(0.33)}\text{Si}_{(2.33)}\text{O}_8$) (pdf 210-8234) which are bracketed in the range (45.20–74.50wt.%). The plagioclases are associated mainly with the Mg-rich pyroxene minerals; enstatite (MgSiO_3), (pdf 900-6339), (37.60wt.% in AZ only); diopside ($\text{CaMgSi}_2\text{O}_6$), (pdf 900-0802), (11.60–17.20wt%) and augite

$((\text{CaFe})_{(0.25)}\text{Mg}_{(0.74)}\text{Si}_2\text{O}_6)$, (pdf 900-9665), (9.90, 21.80wt.% in BA and FA, respectively). These minerals (Fig. 4) in addition to rarely scattered olivine crystals (Fig. 4b) have been confirmed in the microstructure of the basalt samples.

3.2. Pre-melting moduli

The CaO/MgO ratio within the AZ, FA and BA technological samples are 1.51, 1.69 and 0.96, respectively, i.e., (≥ 0.50) (Table 1). Therefore, these technological samples exhibit basic chemical compositions that mitigate their suitability to produce rock wool applying either the duplex or the Cupola technology [5,7,15,27-30].

The high CaO/MgO ratio is due to the low MgO content in all samples. The FA sample is showing the largest CaO/MgO ratio (1.69, Table 1) which is attributed to its mineral contents of both the Mg- and Ca-deriving minerals which are found to be 19.25 and 46.42wt.%, respectively, according to CIPW (Table 2). The maximum CaO/MgO ratio of FA (1.69) would refer to the preferential lower energy required for FA melting and the minimum separation of non-fibrous crystalline olivine or massive amorphous shots during the cinder fiberization when compared with the AZ and BA samples (1.51 and 0.96, respectively, Table

1). The shots and/or olivine would degrade the fiber board quality [5,15,29 and ASTM C1335, 2012].

The calculated M_a values for the technological AZ, FA and BA basalt samples show that all samples meet the high-quality basalt wool characteristics with $M_a \geq 1.80$ recording 3.99, 4.29 and 3.63; respectively, (Table 1). The latter values encourage using the AZ, Fa and BA basalts as main feed for fiber production by the duplex or the Cupola technology [5,15]. In addition, the AZ, FA and BA samples have $K_z \leq 0.70$ with 0.58, 0.54 and 0.60, respectively (Table 1). These values indicate the possibility for all samples to be effortlessly melted at low energy resulting in high molten fluidity with consistent stream flow supporting the easiness of the cinder fiberization [9,30-33].

3.3. Cinders moduli

All moduli of AZ, FA and BA technological basalt samples (Table 1) would suggest their use as main feed to produce wool fiber by the Cupola furnace. Therefore, each sample was mixed with dolomite (Figs. 1b, c) as an additive to formulate the Cupola three feeding raw batches, AZ, FA and BA keeping the same nomination as the raw basalt for simplicity.

After the complete melting of each batch (Figs. 1d, f, Video 1), a cinder sample was picked-up using a steel spoon from Cupola siphon exit (Figs. 1d, f, Video 1). The oxide contents of AZ, FA and BA cinders are falling in the acceptable range values for SiO₂, Al₂O₃, Fe₂O₃, MgO and CaO (43.00-50.00; 6.00-15.00; 3.00-8.00; 6.00-16.00 and 10.00-25.00wt.%, respectively) as deriving cinder for rock wool production (Table 3) [34].

The average CaO/MgO ratio is ≥ 0.50 (1.76, 1.72 and 1.58, Table 3) in AZ, FA and BA cinders, respectively, suggesting consistent melting with high fiberizing ability and fiber quality [5,9,15,30]. The M_a values of all cinders are ≥ 1.80 falling in the range (1.94-2.04, Table 3) with a recorded maximum for FA (2.04) and minimum for BA (1.94). These M_a cinder values (Table 3) are low when compared with their equivalents of the raw basalt (3.63-4.28, Table 1) due mainly to the dolomite charge in the batch samples which would increase the alkaline earth supply. The K_z and M_k values of the AZ, FA and BA cinders are bracketed between 0.66-0.71 and 1.50-2.50 (Table 3), respectively. In addition, the measured cinders temperature found to be 1410, 1350 and 1330°C with a calculated pull rate, i.e., amount of cinder available for fiberization, of 4500, 4200 and 4100kg/h for FA, AZ and BA cinders, respectively. This high pull rate is mainly due to the low surface tension values of the three cinders (404.97,

407.90 and 409.63mN/m for FA, AZ and BA, respectively) calculated based on Kucuk et al. (1999) [38] concerning the cinders chemical composition (Table 3).

It can be concluded that the M_a , K_z , and M_k moduli of the three cinders that bracketed between 1.94-2.04; 0.66-0.71 and 2.06-2.10, respectively, (Table 3), lie in the acceptable range of these moduli. This would encourage the fabrication of flow free surface basaltic wool fibers from the three cinders all with consequent high mechanical properties [4-5,15,30,35-37]. The FA cinder has a temperature of 1410°C, maximum M_a (2.04) in addition to minimum surface tension (404.97mN/m), K_z (0.66) and M_k (2.06) (Table 3). All these values refer to the most stable parameters with economic fiberizing ability of FA cinder during processing (4500kg/h pull rate) [5,9,15]. These optimum characteristics are attributed mainly to the FA cinder higher SiO_2 (46.29) and Al_2O_3 (14.08) over the total alkaline earth oxide contents (29.63wt.%) when compared with AZ and BA cinders (44.82; 13.64; 29.44 and 45.20; 13.40; 30.18wt.%, respectively, Table 3). The lower the mafics, i.e., the Mg-bearing minerals, and the maximum the plagioclases, i.e., the Ca-bearing minerals, of the raw basalt, the optimum is the cinder characteristics for fiber production.

3.4. Characteristics of wool boards

Different wool board samples of densities (50, 70 and 100kg/m³) and thicknesses (100 and 50mm, respectively) have been prepared for AZ, FA and BA to test their mechanical, thermal, acoustic and firing characteristics. The prepared wool boards of density 50kg/m³ will be suffixed by d(50), for example, FA(d50), however, the d(100) samples will be referred without suffix, for example, FA sample. The different density test board samples are pieces of the processed final products (Figs. 2g, h).

3.4.1. Macrostructure

The macrostructure parameters reflecting the single fiber orientation of the wool boards are determined according to Buska and Mačiulaitis (2007) [1]. Figure (5) shows the spatial orientation of the single fibers and surface sections of AZ, FA and BA wool boards showing the longitudinal (L) and the cross (C) fiber orientations either parallel or perpendicular to the board major faces developed as per pendulum movement (Figs. 2b, 5a). The orientation of the single fibers in the longitudinal sections, i.e., S_L (Figs. 5b, c) records 0.64, 0.67, 0.74 in FA, AZ, and BA, respectively (Table 4) referring to the more chaotic fiber orientation in the BA board. On the other hand, the fiber orientations in the cross sections, i.e., S_C , (Fig. 5a) are very close in AZ, FA and BA specimens (0.61, 0.60, 0.60, respectively, Table 4) which might be attributed to the same transverse motion of pendulum during fabrication of all wool boards (Fig. 5a). The S_L values in all

samples are higher than the S_C ones (Table 4) due to the more chaotic fiber orientation at the right and left longitudinal sides (Figs. 5b, c) which are being compressed by the crimping machine during board fabrication increasing the fiber tortuosity (Fig. 2d).

The wool boards are attaining average $S_{L-C} \leq 0.75$ which suggest quasi-horizontal fiber orientation (Table 4) [1,39]. The lowest S_{L-C} is recorded for FA sample (0.62) proposing more planar horizontal fiber orientation in both the longitudinal and the cross directions of FA wool board. AZ and BA recorded the S_{L-C} values of 0.64, 0.67, respectively, which indicate relatively more fiber chaotic orientation in BA (Fig. 5).

The dark and light fiber bands in the wool board samples (Fig. 5) are attributed to the differential distribution of the phenol-formaldehyde organic binder (2.70 wt.%). The dark bands refer to relatively binder-enriched bands when compared with the light ones. The best binder distribution homogeneity is noticed in the order $FA > AZ > BA$ reflecting the relative binder retaining ability on the single fibers. The better the planar structure of the boards with the lowest chaotic fibers, i.e., with the lowest S_{L-C} value, the better is the binder retaining on the single board fibers.

3.4.2. Microstructure

All the board samples are mainly composed of intersecting fibers, shots and pores (Fig. 6 and Table 5). The SEM-EDAX point analyses of AZ, FA and BA board samples (Points 1, 2 and 3, respectively) are shown in table (5) and figures (6d-f). FA fibers are the longest and more continuous (Fig. 6b) than those of AZ (Fig. 6a) and BA (Fig. 6c). The short-broken fibers in AZ and BA would reflect the fiber relative brittleness in these two samples (Figs. 6a, c).

The fiber diameters are bracketed between (2.70-4.10); (5.10-8.60) and (4.30-6.60 μm) in AZ, FA and BA, respectively (Figs. 6a-c). The thickest fiber diameter of FA (8.60 μm) is the main reason for its less brittleness when compared with AZ (4.10 μm) and BA (6.60 μm) (Figs. 6d-f). The thicker the single fiber diameter, $\leq 9.00\mu\text{m}$, the longer and less brittle the fiber is [5,15,40]. The relatively elastic and thicker fibers of FA board are mainly related to the optimum flowability, viscosity and the lower surface tension of their derivative cinder due mainly to the cinder maximum M_a (2.04, Table 3) among all the other samples (Table 3) [5,35]. Moreover, the higher alumina content of FA cinder (14.08wt.%) when compared with AZ and BA (13.64 and 13.40wt%, respectively, Table 3) would improve the elasticity of the derived fibers [8,12,40-41].

The single fibers total SiO_2 and Al_2O_3 contents of FA (61.16wt.%, point 2, Fig. 6e, Table 5) are higher than those of AZ and BA (57.59, 50.13wt.%; points 1, 3; Figs. 6d, f, Table 5, respectively). This is in conformity with the least alkaline earth, MgO and CaO, summation for FA (27.25wt.%, point 2, Fig. 6e, Table 5) compared with AZ and BA single fibers (29.49 and 32.75wt.%; points 1, 3; Figs. 6d, f, Table 5, respectively). These oxide concentrations of single fibers are in conformity with their concentrations in the deriving cinders (Table 3).

The observed lemon, spandrel and potato-like particles are the non-fibrous glassy granules known as “shots” (Figs. 6g-i). The shots of each sample were mechanically separated according to ASTM-C1335, where each loose fiber sample was brushed using the sieve set Nos. 20, 50 and 100. The total shot contents are 13.00, 14.00 and 16.00wt.% in FA, AZ and BA boards, respectively. These contents are in the acceptable range ($< 25.00\text{wt.}\%$) of the insulation board according to ASTM-C1335.

The minimum shot content in FA board (13.00wt%) could be mainly attributed to its cinder maximum M_a (2.04, Table 3) causing the optimum melt flowability (Table 3) [7]. The SEM-EDAX analysis shows that the summation of SiO_2 and Al_2O_3 of FA shots (58.29wt.%, point 5, Fig. 6h and Table 5) is relatively higher than in AZ (57.91wt.%) and BA (57.90wt.%) (Points 4, 6; Figs. 6g, i; Table 5,

respectively). On the contrary, the summation of the crystalline alkaline earth oxides is the lowest in FA shots (27.25) (Point 5, Fig. 6h; Table 5) and the maximum in AZ and BA (29.79wt.%) board samples (Points 4, 6; Figs. 6g, i; Table 5, respectively). These oxide concentrations are would explain the relative enrichment of AZ and BA with shots compared with FA sample. The compositions of shots and their derivative cinders are conformable (Tables 5 and 3, respectively).

3.4.3. Pore system

All board samples are showing pores because of the fiber intersections (Fig. 6). The apparent porosity of the board samples determined by Helium porosimeter records 95.28, 95.51 and 95.07% in AZ, FA and BA samples, respectively.

The summary statistics for X-ray μ CT analysis of the wool boards is presented in Table (6). A 2D slice of each analyzed volume is shown in Figs. (7a, d and g) whereas the 3D rendering images of the fiber structure and pore space local diameters are shown in Figs. (7b, e and h) and (7c; f and i), respectively.

The visible porosity obtained by the μ -CT (Table 6) is consistently lower than the values obtained with Helium porosimeter either due to the limited image resolution of the tomography (9 μ m) or the non-representative small analysed

volume for each sample (Table 6). However, the order from low to high porosities (BA < AZ < FA) is identical for both measurement techniques. In all board samples, the porosity is very high which leads to perfect pore connectivity (>0.99) (Table 6), except for few air inclusions in the shots. Likewise, the pore tortuosity is negligible in all samples due mainly to their perfect connection. The most frequent fiber diameter is equal in all samples and amounts to $30\mu\text{m}$. However, the SEM analysis proves the fiber diameters to be bracketed between $(2.70\text{--}4.10)$; $(5.10\text{--}8.60)$ and $(4.30\text{--}6.60\mu\text{m})$ in AZ, FA and BA, respectively (Figs. 6a-c). The difference of the fiber diameters is mainly attributed to the limited resolution of the tomography ($9\mu\text{m}$) and the chosen segmentation threshold that was optimized for retrieving fiber continuity on the expense of diameter overestimation that also resulted in an underestimation of visible porosity.

The CT analysis shows that the AZ wool board sample has the highest shot volume fraction (3.40%, Table 6). This could be attributed to the AZ cinder high content of CaO as a crystallizing agent (18.76wt.%, Table 3) which would minimize the AZ cinder flowability [5,15,30,34-35]. As a result, the AZ board sample has the lowest fiber density ($82\text{mm}/\text{mm}^3$, Table 6) with a consequent highest average pore diameter (0.20mm , Table 6). The BA and FA samples have visually a similar microstructure (Fig. 7). A higher sample number would have

been required in order to reach a more robust quantitative differentiation between the two wool board types. This lack in representativeness is of the small sample volumes is indicated by the mismatch between shot content (mass wt.%) in CT images (4.8-39.2wt.%, Table 6) and the mechanically separated according to ASTM-C1335 (13-16wt.%, Table 7).

3.4.4. Mechanical characteristics

The average compressive strength values are 10.47, 13.35 and 14.29kPa at 104.73, 133.52 and 142.88N, for BA, AZ and FA insulating boards, respectively (Table 7). The former compressive strength values are in the acceptable range adopted by EN 13162 and ASTM C612 (≥ 0.25 and ≥ 2.40 kPa, respectively). The point load values of FA, BA and AZ are 140, 130 and 100N, respectively (Table 7). All the latter values are agreeing perfectly with EN 13162 which typifies the accepted point load to be ≥ 50 N. On the other hand, the obtained tensile strength is 3.00, 2.50, 2.00kPa recorded for FA, BA and AZ, respectively (Table 7) which lie in the acceptable range of EN 13162, i.e., >1 kPa (Table 7). All the mechanical characteristics of the present work boards encourage their utilization in the different load bearing applications according to the EN and ASTM standards.

The FA is showing the optimum mechanical characteristics when compared with the AZ and BA board samples. This could be interpreted in terms of its macro- and microstructure. The lowest S_{L-C} value of FA board (0.62) proposes better quasi-horizontal fiber orientation when compared with AZ and BA boards (0.64 and 0.67, respectively) with relatively lower affinity planar structure (Table 4). Such dominant fibers orientation in FA proposes their perfect near perpendicular orientation against the applied load with the least chaotic fibers tending to be less elastic and easily deformed [42]. On the other hand, the FA quasi-horizontal orientation encourages the well distribution and retention of the phenol-formaldehyde organic binder (Table 7) among the board single fibers which consequently add to the resistance to any applied load [8,42]. The quasi-horizontal fiber orientation of FA diminishes the retarding effect of its optimum porosity of (92.30%), moderate pore diameter values (0.16mm) and fiber length density (123.00mm/mm^3) (Table 6, Figs. 7d-f) on its optimum mechanical characteristics among BA and AZ board samples (Table 7).

In addition, the microstructure of FA board sample shows relatively longer and thicker fibers ($5.10\text{-}8.60\mu\text{m}$) (Fig. 6e) when compared with its equivalent AZ and BA of shorter single fibers and relatively smaller diameter ($2.70\text{-}4.10$ and $4.30\text{-}6.60\mu\text{m}$, respectively) (Figs. 6a, c). The length and diameter of the single fibers are in direct relation with their derivative cinders flowability behavior

which is optimized in the FA cinder (Table 3). The longer and thicker the board single fibers, the minimum brittleness and consequently the better is its resistance to any applied loads [4,40-41]. Additionally, the FA board is of the least shot contents (13.00wt.%) (Fig. 6h, Table 7) which could be considered as harmful-microstructure bodies to interrupt the continuity of the single fiber orientation (Figs.7b, e, h). Shots are points of weakness for the mechanical characteristics in general, therefore, the lower the shots content, the better is the mechanical characteristics of the wool board (Table 7) [42-44].

The present work wool board samples have compressive strength values ≥ 10.00 kPa. This may be attributed to the better fibers orientation of the present work wool board samples attaining quasi-horizontal planar orientation with $S_{L-C}=0.64$, 0.62 and 0.67 of AZ , FA and BA, respectively, compared to the quasi-chaotic fibers orientation ($S_{L-C}=0.76$) of Buska et al., (2015) [39] wool board.

3.4.5. Thermal characterization

The d(50) and d(100) wool board samples with thicknesses 200.00 and 50.00mm, respectively, are characterized for their thermal properties by measuring K- and R-values using lambda meter “Guarded hot plate type” based on EN 12667 test method (Table 8 and Fig. 8a).

The K-values of d(50) and d(100) are bracketed between 32.43 and 36.00mW/mK (Table 8). These are in the acceptable K-values range for the wool boards according to EN 13162, EN 14303, ASTM C612 and GB/T 25975 (≤ 60 , ≤ 65 , ≤ 36 and ≤ 40 mW/mK, respectively, Table 8, Fig. 8b). On the other hand, the measured R-values for the d(50) and d(100) boards fluctuate from 1.39 to 6.17m²K/W. The R-values of d(50) boards are bracketed between 5.95 and 6.17m²K/W which lie in the acceptable range for the insulating wool boards according to EN 13162, GCCC specs for walls, GCCC specs for roofs, ECBC specs for walls concerning 24h use building, ECBC specs for roofs concerning 24h use building, ECBC specs for walls concerning day use building and ECBC specs for roofs concerning day use building (≥ 0.60 ; ≥ 1.35 ; ≥ 1.75 ; ≥ 2.10 ; ≥ 3.50 ; ≥ 2.10 and ≥ 2.10 , respectively). On the other hand, the R-values of d(100) are bracketed between 1.39 and 1.41m²K/W which are only acceptable according to EN 13162 and GCCC specs for walls (≥ 0.60 ; ≥ 1.35 m²K/W, respectively). The K- and R-values of the d(50) and d(100) of the AZ, FA and BA are acknowledging their possible use as perfect thermal insulating boards for buildings roofs and walls reducing the heat transfer in the summer and winter achieving thermal comfort.

All the insulating boards of (d50)-200mm have lower K-values and higher R-values, than those of (d100)-50mm in all samples (Table 8). This could be due

mainly to the larger volume of the entrapped-air among the available pores of the low-density (d50) board samples compared with the entrapped-air volume in the high-density (d100) boards [45].

The FA(d50) and FA(d100) boards recorded the minimum K-values (32.43 and 35.35mW/mK, respectively) and the maximum R-values (6.17 and 1.41m²K/W, respectively, Table 6.10), i.e., FA boards are of better thermal insulation when compared with the AZ and BA. This could be attributed to the higher porosity values (92.30) of FA when compared with those of AZ (91.30) and BA (88.40%) board samples (Table 6 and Fig. 7). On the other hand, the porosity values of the wool boards confirm that the solid part of each board, i.e., the fibers and shots, would be 7.70; 8.70 and 11.60wt.% in FA, AZ and BA, respectively.

The thermal conductivity of the wool board is affected by the single fibers chemical composition. It is proportionally increasing with the iron and titanium and decreasing when silica and alumina are relatively higher [16, 20]. The microchemistry of the FA fibers (5.10-8.60μm) shows that the total thermal conductivity initiating oxides (CIO), Fe₂O₃; TiO₂; CaO and MgO (34.98wt.%) is the least when compared with the CIO of AZ and BA (39.09 and 46.55wt.%, respectively, Table 5). In addition, the total thermal conductivity ceasing oxides (CCO), SiO₂; Al₂O₃; Na₂O and K₂O (65.02wt.%) is the maximum when

compared with the CCO of AZ and BA (60.91 and 53.46wt.%, respectively, Table 5). Therefore, the single FA fibers would minimize the heat transfer through them enhancing the whole thermal resistance of the FA board. The microchemistry of the shots is in conformity with the fibers in each board sample (Table 5).

The present work wool boards of density 50-100kg/cm³ are of better K-values (32.40-36.00mW/mK) when compared with those of 13-241kg/cm³ and 38.00mW/mK of Domínguez et al., (2009) [45]. The lower density wool boards have higher entrapped-air among their pores to assure better K-values [45].

3.4.6. Fire resistance

The rock wool boards are inorganic non-combustible insulations and can withstand temperatures without gas evolution when compared with the organic ones such as foam and polyurethane [46-48]. The studied rock wool boards can withstand temperatures above 1000°C (Fig. 8) without gas evolution. The fire performance parameters in terms of the calorific value (PCS, MJ/Kg); the mass loss upon heating (Δm , wt.%); the furnace temperature increase (ΔT , °C); the duration of flaming (T_f , sec); the flame growth (FSI) and the smoke concentration (SDI) are measured for FA, AZ and BA insulating boards

according to EN ISO 1716; EN ISO 1182 and ASTM E84 (Table 8). The results obtained prove that FA, AZ and BA can be used as fire resistant insulating boards. The boards fire parameters (Table 8) are matched with EN 13501-1 and found to perfectly conform to the “European A1” fire behavior and “class A” of ASTM E84.

3.4.7. Acoustic characterization

The wool boards of FA, AZ and BA with variable densities but same thickness were examined for acoustic insulation by the determination of their noise reduction coefficient “NRC”, sound absorption coefficient “ α_s ” (Fig. 8) and sound transmission class (STC) according to the European Standard EN ISO 354, ASTM E90 and ASTM E-2235, respectively [49-51]. All board samples have α_s of 1 (Fig. 8d), however, the AZ(d100) and FA(d70) have 1.05 NRC whereas the BA(50) has 0.95 NRC. These values prove the excellent noise absorption of all boards according to ASTM C423 and found to be categorized as “Class A” noise absorption class according to EN ISO 11654.

The high NRC and α_s values of all samples could be attributed to the tortuous (1.01, Table 6), interconnected and channeled pores (>0.99%, Table 6) generated by the intersection of single fibers in each board sample (Figs. 6a-f and Figs. 7b, e, h). These pores enhance the noise absorption by increasing the path distance

of the sound waves which move among the fibers in all samples (Figs. 6a-f, Fig. 7c, f, i) [52-54]. Consequently, sound waves are damped through the wool board and attenuated by the multiple reflections till completely degenerated into thermal loss [55-56].

Zhu et al. (2015) [50] reported that different non-woven wool boards in the density range of 30-90kg/m³ have 0.02 to 0.98 α_s values which are better than the α_s values of Hua and Yang (2018) [57] (0.09-0.77) for samples having the same density. Nevertheless, the present work wool board samples still achieve the best α_s values (0.10-1.15).

4. Conclusions

1. The optimum pre-melting and cinders moduli of the investigated basalts are attributed to the minimum concentration of Mg-bearing minerals.
2. The higher the basalt cinder viscosity/acidity modulus to surface tension, the optimum is the economic fiberizing ability during processing (4500 kg/h pull rate).

3. The mechanical characteristics of the wool boards have been optimized to record 28-, 3- and 2.8-times the compressive and tensile strength as well as the point load promoted by the EN standards.
4. The dominant fiber orientation style, single fibers diameter and optimum alumina content have the major impact on the wool boards mechanical characteristics rather than the single fibers length density and tortuosity as well as the porosity and pore connectivity of the wool boards.
5. The thermal insulation characteristics of the studied wool boards are inversely related to the single fibers concentration of conductivity initiating oxides. The thermal characteristics in terms of K and R are improved 1.30- and 2.35-times more than the values adopted by the EN standards.
6. All wool boards have high NRC and α_s to be categorized as “Class A” noise absorption class. This is attributed to the tortuous, interconnected and channeled pores generated by the intersection of fibers in each board. These pores increase the path distance of the sound waves and consequently damp and attenuate the sound waves. The acoustic characteristics in terms of α_s and NRC have been improved 1.10- and 1.40-times more than the EN standards.

7. All the engineered wool boards have superior fire resistance with recorded SDI (0) and FSI (0) when compared with the ASTM standards (450.00) and (25.00), respectively.

5. Compliance with ethical standards

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Video Captions

Video 1: Video showing the fiberization process of FA cinder in Cupola furnace.

Video 2: Video showing the whole process of wool board manufacture.

Figure Captions

Figure (1): Photomicrographs showing the batch melting and hot end fabrication process of wool boards. (a, b) The raw basalt and dolomite in the storage yard; (c) The charging of raw mix basalt and dolomite into the Cupola; (d) The basalt cinder pouring out of the “siphon” exit; (e) The collection of cinder sample by a cinder spoon; (f) The cinder temperature is measured by cinder pyrometer; (g) The fiberizing machine with the rotating fiberizing discs containing the binder distribution nozzles; (h) The hot single fibres exit the rotating fiberizing discs.

Figure (2): The cold end fabrication process of wool boards. (a) The collection of single fibres on a rotating drum sheet by suction; (b, c) The lateral fibre distribution by pendulum into fibre mattress; (d) The compression of fibre mattress by crimping machine before inlet to a curing oven; (e) The fibre mattress after exit from the curing oven; (f) The longitudinal cutting “slitting” of

wool boards to cut the required width; (g, h) The boards conveyed to the stacking machine for final packing.

Figure (3): XRD diffractograms of AZ, FA and BA basalt technological samples.

Figure (4): X-Nicole's photomicrographs showing the microstructure and mineral content of AZ (a), FA (b) and BA (c) basalt technological samples. In all samples, augite (Au) is surrounded by plagioclase (Pl) mineral crystals.

Figure (5): Photomicrographs showing the macrostructure of AZ wool board. (a) 3D arrangement of fibre in AZ wool board showing the pendulum movement (black arrow); the cross "c" and the longitudinal "L" sections; (b) The fibre orientation at the left side of AZ wool board; (c) The fibre orientation at the right side of AZ wool board.

Figure (6): SEM-Photomicrographs showing the microstructure of wool boards. (a-c) SEM-photomicrographs showing the fibre morphology of AZ, FA and BA wool boards, respectively; (d-f) SEM-photomicrographs showing the fibre diameters and point analysis positions (1-3) of AZ, FA and BA wool boards, respectively; (g-i) SEM-photomicrographs showing the shots morphology and point analysis positions (4-6) of AZ, FA and BA wool boards, respectively.

Figure (7): Photomicrographs showing the pore system of the studied wool board samples. (i) μ -CT 2D slices of BA, FA and AZ analyzed volume (a, d and g, respectively); (ii) μ -CT 3D rendering fibre structure images of BA, FA and AZ (b, e and h, respectively) and (iii) μ -CT 3D rendering pore space diameter images of BA, FA and AZ (c, f and i, respectively).

Figure (8): The thermal, acoustic and reaction to fire characterization of wool boards. (a) The thermal conductivity (mW/mK) vs time (minute) graph of FA(d50) wool board; (b) The thermal conductivity (mW/mK) and resistance ($\text{m}^2\text{K/W}$) values of the wool boards; (c) The sound absorption coefficient (α_s) vs the frequency (Hz) of the wool boards; (d) The AZ wool board before fire resistance test; (e) The AZ wool board after the fire resistance test as seen from the exhaust end.