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# Influence and Application of Glass Cullet in Autoclaved Materials

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**Abstract.** The basis of construction is made of materials such as: wood, stone (including artificial stone, i.e. concrete and brick), steel and glass. Currently, the glass is a product that enjoys increased interest, also due to the phenomenon of recycling. At the moment, construction industry is a field in which good solutions are sought for the proper management of waste. One of the recycled waste is glass cullet coming from bottles mainly of wine. Not every country produces wines because there are no suitable conditions, but wine is drunk in every country. Powdered glass is already applied in the form of glass powder in concretes (research conducted in India, China and America), and the results of the research are the basis for exploring this topic in the context of other materials. The paper presents the results of research on modifications of sand-lime materials using cullet (glass sand). Non-destructive testing of samples was carried out: lime hydration control in a mixture containing powdered recycled glass, /humidity that affects the water absorption of the product, and further on density and strength (water migration in the pores) and microstructural investigations. The conducted tests showed increased humidity of the modified product with recycling glass (approximately 2% compared to the reference sample of 0.5%) and the presence of hydrated calcium silicates in the form of gyrolite as a phase resulting from high pressure.

## 1. Introduction

Glass is a material commonly used in the world in virtually every economic sector. Production, use and subsequent utilization of glass in the form of car windows, window frames, glazes of all forms, bulbs or alcohol bottles is such a big problem as the disposal of car tires. In the era of balanced development, care for the environment, overproduction, which is associated with the so-called Economic and energy responsibility are sought for alternatives to solve the problem. A popular way to use glass cullet is to grind it to the micro fraction and to use the resulting component for the production of concretes [1, 8, 9,11]. Another option of combating the surplus of glass being recycled is the use of milled glass for small fractions in autoclaved materials, such as sand-lime bricks. These materials are made of natural components such as silica (SiO<sub>2</sub>) of quicklime quenched before being mixed with sand and water, and undergo a hydrothermal treatment at a temperature of about 200°C and pressure of 1.2-1.6 MPa. The article presents the method of using cullet in autoclaved materials and describes the scheme of laboratory tests (cullet in the form of glass sand (GS)). The main attention was focused on changes occurring in the structure of the internal modified material and its microstructure. Moisture and microscopic analysis (SEM-scanning electron microscope and XRD) were carried out to determine the interaction between glass sand and lime particles, which affects the distribution of pores and other physico-mechanical properties of the discussed materials.



## 2. Methodology and laboratory tests

Laboratory tests were carried out to verify the correctness of the proposed modifications [12,13,14,15]. Quartz sand, quicklime and glass cullet were used for research. Glass sand comes from the recycling of bottles. The bottles are suitably selected, soaked and washed to remove residues that could enter into the reaction with any of the components of the mass. Bottles are also segregated due to the colour, because, as proved by analyzes and research [8], the colour affects the properties of the final product and the course of chemical reactions occurring in the production of individual materials. In the mass, lime CaO was used in the amount of 7% and in the hydration process it was mixed with 5% water and quartz and / or glass sand (90% GS was the total elimination of quartz sand (SiO<sub>2</sub>)).

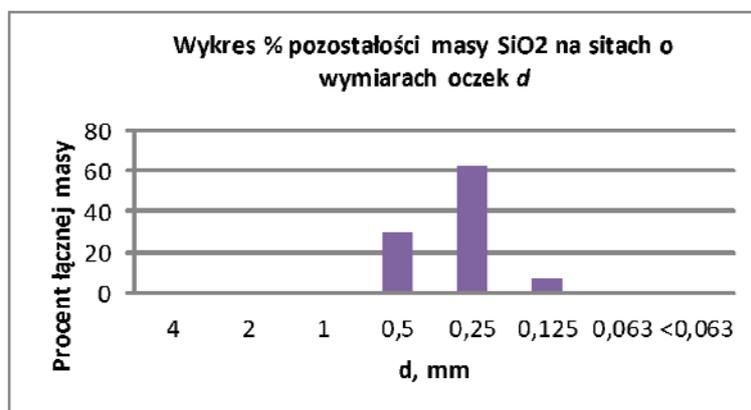
**Table 1.** Percentage of modifier in relation to mass product [%]

No	CaO	SiO <sub>2</sub>	GS
1	7	90	-
2	7	80	10
3	7	70	20
4	7	60	30
5	7	50	40
6	7	40	50
7	7	30	60
8	7	20	70
9	7	10	80
10	7	-	90

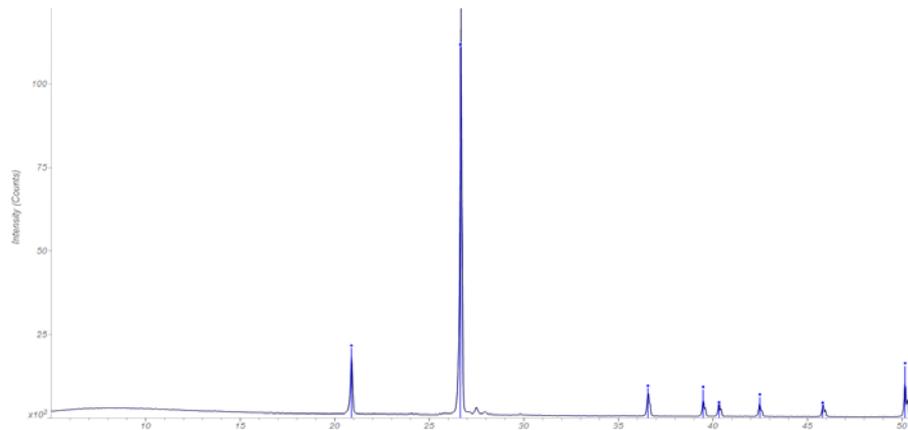
### 2.1. Analysis of the composition of autoclaved materials

Most of the samples for analysis were made in LUDYNIA (Bricks Production Company belong to Silicates Group, Poland) and some of the samples were made during an internship at UdeS in Canada. Material samples in the amount of 12 were made for preliminary analyzes (6 samples with dimensions 5x5x5cm and 6 samples with dimensions 4x4x16 cm). The tests were conducted on 2 types of samples to examine the dependence of the mass distribution in the proposed samples (mixing, stacking, compacting and autoclaving of the products). The results in both cases were convergent.

Quartz sand was used for analysis (Figure 1,2). It was sand with small admixtures of aluminium and, the analysis shown that the size of the grain of size did not exceed 1mm, while the most shares had grain in the range (0.125-1mm).



**Figure 1.** Sand residues on sieves of mesh size 'd'.



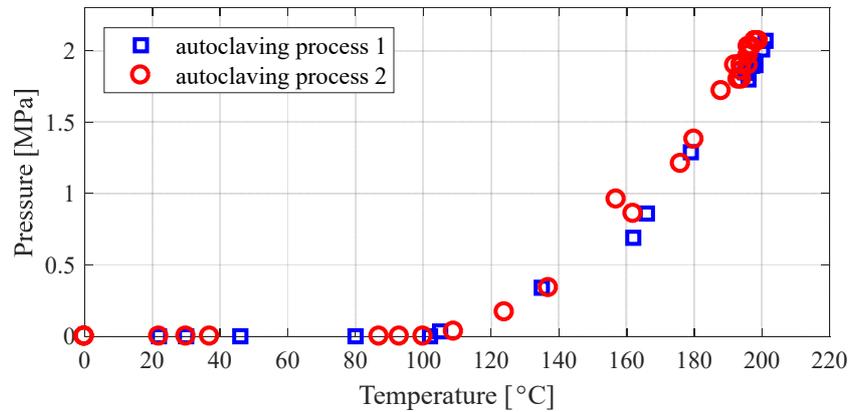
**Figure 2.** XRD for quartz sand.

*2.1.1. Hydrothermal conditions and laboratory tests.* The diagram of the laboratory autoclave is presented below. The working time was 5 hours. Samples remained in the autoclave until the device cooled down, and thus the pressure drop, due to work safety. In the industrial production of bricks by autoclaving, leaving the material in the autoclave to cool down reduces the risk of damage to the internal structure of the material (which may occur in the case of fast contact between the fired product and the cold air outside the autoclave). The table shows the working time of the autoclave from the moment the device was turned on until the next day of laboratory samples were removed from it (table 2).

**Table 2.** Dependence of pressure on temperature during autoclaving of laboratory samples.

No	Time [h]	Temperature [°C]	Pressure [MPa]
1	(Start, 14.30/2.30 p.m.)	22	0.00
2	15.00	90	0.00
3	15.05	100	0.03
4	15.30	162	0.76
5	15.45	198	1.90
6	16.00	200	1.97
7	16.30	197	1.93
8	17.00	198	1.90
9	17.30	197	1.80
10	18.00	200	1.93
11	18.30	197	1.80
12	19.00	196	1.72
13	19.30 (the end of the autoclave work)	201	1.93
14	21.00/9 p.m.	161	0.69
15	Next day, 8.00 a.m. (opening the autoclave)	34	0.00

In each of the tests, 12 samples of the same composition and under the same conditions were made. The process and temperature of hydration of lime were controlled, the mass quality with the modifier and the conditions of autoclaving. Due to the potential of the autoclave, the autoclaving time was 5 hours each time. The graph below shows an example of the autoclave test (Figure 3).



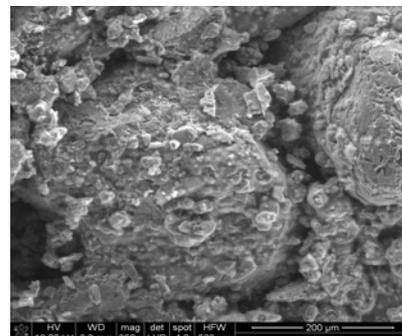
**Figure 3.** Pressure vs. temperature dependence of autoclave processes

**3. Microstructure of modified autoclaved materials**

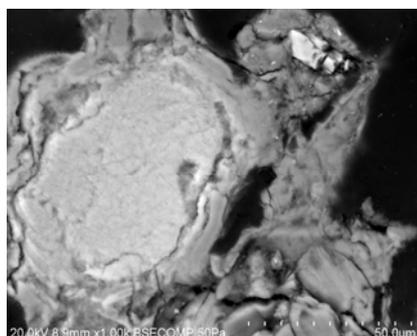
Materials formed in hydrothermal conditions and with the participation of high pressure are characterized by the presence of calcium silicates hydrated. While the C-S-H or tobermorite phase is typical for traditional sand-lime autoclaved materials, a special case is the presence of xonotlite or giolite. Research has shown that the more structured internal structure of the material, the greater their surface area [10] which changes, inter alia, the severity, durability and strength of the materials tested (Figure 5-11). Due to the presence of Na in the GS-modified material, a new phase is observed, which is gyrolite (Figure 9).



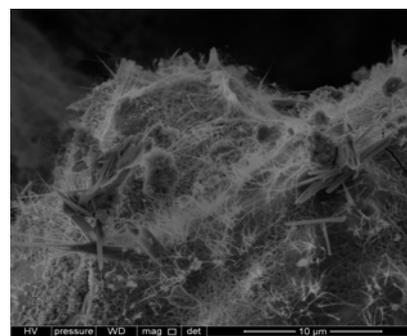
**Figure 4.** Traditional sand-lime brick. Broken off part of the material.



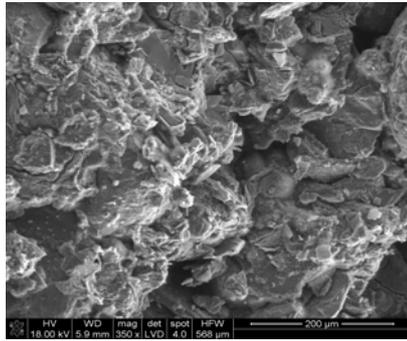
**Figure 5.** The sample of a traditional brick(SEM).



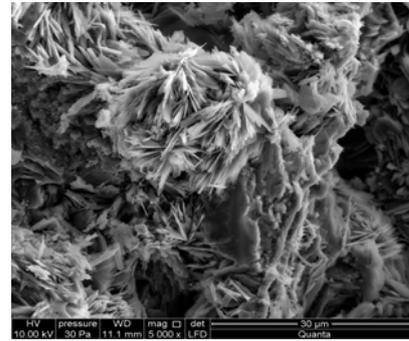
**Figure. 6.** 50% SiO<sub>2</sub> & 50%GS (low vacuum).



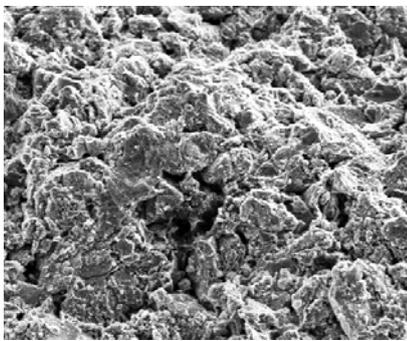
**Figure 7.** 50% SiO<sub>2</sub> & 50% GS (high vacuum.)



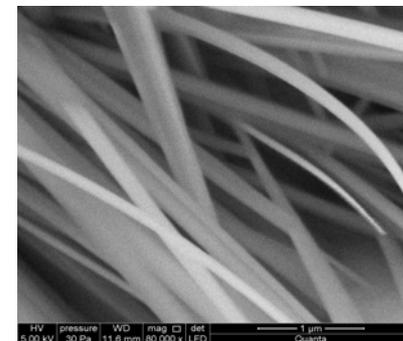
**Figure 8.** 90% GS (high vacuum).



**Figure 9.** 90% GS (after 1 year).  
Products with glass cullet (gyrolite  
C/S=0,67).



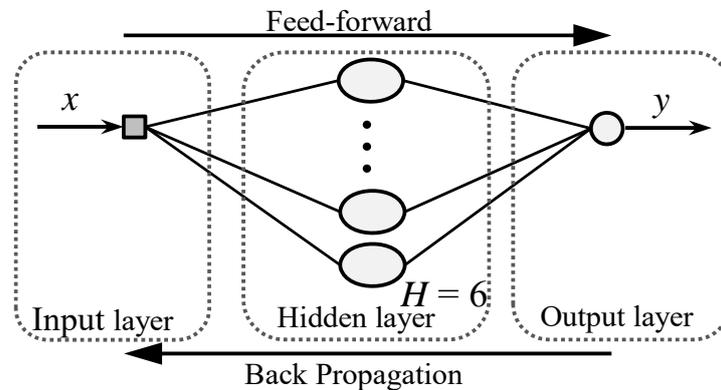
**Figure 10.** 90% GS (after 1 year, high  
vacuum).



**Figure 11.** 90% GS (after 1 year, high  
vacuum).

#### 4. Research and prediction of moisture of sand-lime bricks using neural networks

Artificial Neural Network (ANN), due to their mode of action, are very effective in the analysis of the problem of prediction. In the paper a Backpropagation Neural Network (BPNN) was applied. This type of network is often presented as a universal approximator capable of modelling of the function with any complexity law. Modelling using PBNN, is an iterative search of a non-linear relation in considered model, using the given data set, without the necessity to give assumptions of concerning the structure of model [2, 3, 4]. The determining the properties of sand-lime materials will be predicted on this research by using neural networks. ANN method was chosen because this method can be used as a model to develop tools to predict the moisture and other properties of this kind of materials, and can anticipate the nonlinearity and the complex interactions between input and output variables of this kind of bricks [7]. Before starting the prediction, is necessary to determine the number of hidden layer nodes and weights of the ANN which will be used. To determine the matter, ANN training on variation of hidden layer nodes between the nodes 1-10 was conducted to determine the most accurate ANN. The parameters used to determine the best ANN is the mean square error (MSE) of training and validation. After the preliminary calculations, the network architecture was accepted (BPNN: 1-6-1), for a further analysis (Figure 12).



**Figure 12.** Architecture of BPNN: 1-6-1

The log-sigmoid transfer function: was assumed for hidden layer neurons and linear function in the output layer. Defining the network was carried out off line using Neural Network Toolbox, working in the MATLAB computing environment [6]. For the learning the Pseudo-Gaussian Levenberg-Marquardt method was applied.

In the analysed task, to identify moisture of the material, the network was formulated with the samples obtained from laboratory tests. 40 learning and validating patterns and 20 patterns for the testing of PBNN network were extracted. During the identification, the input  $x$  to the network was the percentage of Glass Sand (GS), whereas the network response  $y$  was the moisture of the material  $W$ .

In order to compare the efficiency of the BPNN network for the prediction of the material parameter  $W$ , a traditional approximation of the obtained results was also made. The following form of the function has been developed (1):

$$f(x) = 0.491 + 2.322 \sin(x), \quad (1)$$

The coefficients of the function which approximating the  $W$  (GS) diagram were determined using the least-squares method, while the selection of the combination of the base functions was carried out using the "trial and error" method. The Table 3 contains the maximum values of absolute error -  $e$ , mean square error -  $MSE$  and the linear regression coefficient -  $r$ , which will allow to estimate the quality of the performed neural network prediction and classical approximation. Figure 13 shows the correlation of  $W$  values calculated using neural networks and classical approximation. This correlation is close to unity in both cases (Table 3).

**Table 3.** Errors of approximations.

Procedure	$\max e$ [%]	$MSE$	$r$
BPNN :1-6-1	12.23	0.006	0.970
Approximation	14.02	0.026	0.965

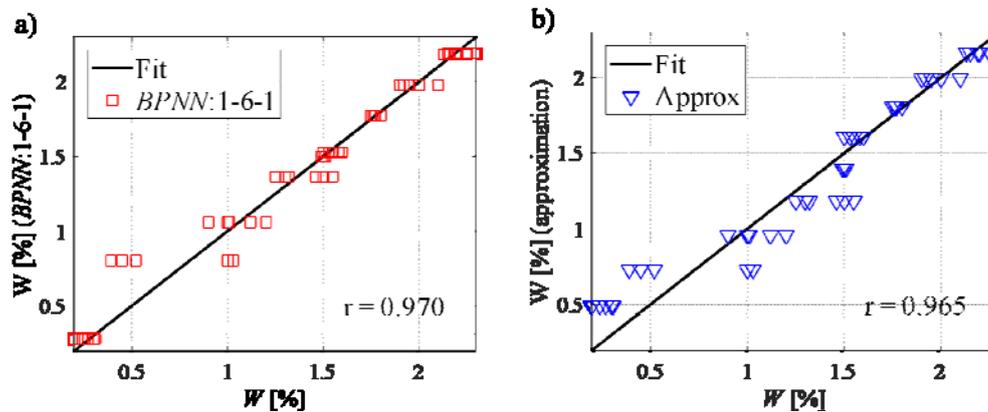


Figure 13. Regression plot of a) BPNN prediction and b) approximation.

On the basis of the results presented above, it can be concluded that both procedures were realized correctly. Moisture of the material estimation errors are smaller when using a neural network. Research has shown that the increase in the share of glass cullet (GS) in mass, increases the moisture content of the material after autoclaving process (Figure 13,14).

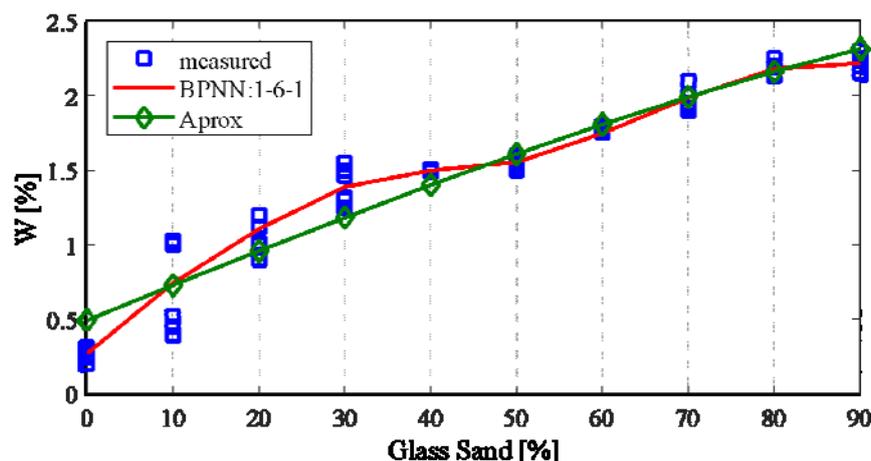


Figure 14. Simulation results of the effect cullet glass (GS) on moisture of the modified material.

## 5. Summary and discussion

The use of glass components (cullet glass) changed the humidity/moisture of the modified brick (the humidity of this type of brick increased by 1.5%). It is concluded that the absorbability of the material will be reduced. In the further part of the analyzes, the following tests should be performed: compressive strength, bulk density, XRF, XRD of the modified material, DTA and water absorption). In the paper of Hornik [5], it was proved that Multi-Layer Perceptron (MLP) networks with three layers can be theoretically assent as universal approximators. Many works indicate that neural networks are equal to or even surpasses the classical methods related to the estimation of statistical models and prediction. The rationale for the use of neural networks for the prediction of material parameters is their simpler implementation in expert systems, connected, for example, with research equipment (microscope, autoclaves, presses for compressive strength tests), [3].

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