RESEARCH ON THE RECOVERY OF SOME AGRICULTURAL WASTE FOR THE MANUFACTURE OF COMPOSITE MATERIALS WITH CLAY MATRICES

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Abstract: The article describes the results of the research carried out for the systemic description of the manufacturing technological processes of composite materials with clay matrix and insert from agricultural waste. The objectives of these researches include the valorization of agricultural waste and cheap and abundant resources to obtain composite materials usable in civil constructions or various rural developments, with properties superior to existing ones, for example adobe or clay bricks. The article describes the materials used, the technology of obtaining, the dependence between the output parameters and the input and control parameters.

Keywords: agricultural waste, composites, clay, technology

1. INTRODUCTION

According to [1], composite materials are a new class of materials of great technological importance; they are systems of deformable solid bodies, macroscopic-scale combinations of several materials. In short, composite materials are also called composites [2]. According to the technical and scientific definition, according to [2], materials that bring together in a single product some elements that usually do not associate naturally, they are called composite material. According to [3], composite materials emerged as a substitute for classic materials in order to offer manufacturers other raw materials, with superior properties, that are easier to produce. Today these raw materials are present in all fields of activity thanks to the fact that they can be "designed" individually according to the properties required by the final destination of the products to be made. By designing them according to the requirements, the properties of these raw materials are clearly superior to those of each component taken individually acting that synergy similar to teamwork. This solution, which leads to the achievement of the producers' wishes, however, also has negative aspects, as part of the plastic components in their structure are not degradable and are not environmentally friendly. Also, in [3] it is stated that composite materials are present in nature since time immemorial: wood, soil, bones of the human body and other beings. The earliest composite materials used by humanbeen were straw and mud used to make building bricks [4]. Other examples of composite materials from various eras of human existence are given in [3]. Some of the oldest building materials are adobe [5], defined as a brick-shaped building material made from a mixture of clay, straw and dung dried in the sun. Many houses in human history have been built using strong wooden and adobe structures.

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In [6] it is shown that the building standards today have reached a level that is difficult to imagine a few decades ago, but a careful look at the construction method and the advantages of adobe houses is of particular interest even nowadays. This solution has its roots far back in time, being adopted in most regions of the globe. The advantage of obtaining the main building material and its heat-insulating qualities is the basis of this interest.

In [7], the special qualities of adobe are stated: "Although it is considered that adobe homes are specific to poor categories of the population, some specialists admit that they seem to be the healthiest houses and, at the same time, the most resistant to earthquakes. Adobe is a good thermal insulator, receiving heat with difficulty and giving it up slowly. The clay and straw that form the basis of the construction of such houses are ecological, natural materials, providing a healthy indoor environment".

Classic adobe recipes [8-10], involve mixing clay with horse dung and straw, forming and drying in the sun. In contrast to the adobe recipe, the recipe for obtaining composite materials proposed by the authors is ultimately based on compression of the composite mold, which also achieves additional dehydration, then natural or forced drying (dehydration). Forced compression gives it greater hardness than adobe.

There are numerous articles reporting research on matrix composite materials from agricultural waste soils and fills [11-18]. The mechanical characteristics of composites of this type are the subjects of some articles [19-23]. Samples of clay bricks are presented and examined by the axial compression test, Figure 4 of [23]. In [24] the authors present a series of physical and mechanical analyzes of mud bricks mixed with recycled asphalt. The authors [25] analyze the thermal insulation properties of mud bricks reinforced with glass fibers.

Mechanical properties of the same material are studied by the authors [26, 27], for various clay concentrations. Affirming this type of materials as sustainable materials, the authors [28, 29] highlights their thermal properties. Coconut fibers are also tested as reinforcement materials in cement bricks or mud, [30]. Reinforcement with straw is reviewed, for example, in [31], and that with sisal fibers in [32]. The mechanical properties of bricks reinforced with sugar beet fibers and bioenzyme reinforcements are presented in [33].

Numerous authors analyze the composites obtained from a matrix material with several types of insert materials [29]. Apart from waste materials of various types, many authors test optical fibers [34].

Numerous works analyze the manufacturing technologies of bricks or briquettes of composite materials with a base of clay and fibers of various types [35-37].

The main objective of the research described in this article is the characteristic for the preliminary loadings necessary in the design of a product and its manufacturing technology. For this reason, the main objective of these researches is to determine if the realization of the material and a technology to obtain it is feasible. To achieve this objective, a series of secondary objectives can contribute to the configuration of the work methodology in deep research. Among these objectives, we mention:
- defining the composite material variants (matrix material, potential candidate materials for inserts, materials used in the manufacturing process).
-inventory of input, output and order parameters in the manufacturing process.
-estimating the existence of possible links between the parameters selected for the previous objective.
- the selection of a number of output parameters that, in quantified form, can estimate the performance of the manufacturing process (consumption, yields, lubrication) and the performance of the material produced (mechanical resistance, thermal insulation capacity, physical stability over time, etc.).
- fixing some working intervals in the research activity, for input parameters and order (concentrations of inserts in the material, humidity in the compression-forming process, working temperature).

2. MATERIAL AND METHODS

The approach to the study of these materials is made from at least two points of view. The first is obviously the manufacturing technology of the material or materials. The second point of view refers to the physical properties that the materials thus obtained have. Thus, in the materials chapter we refer to the physical and chemical properties of the component materials of the composite and to the technology of obtaining them, and in the methods part, we can partially include an exposition about possible technological options, but especially the test methods that must highlight the physico-chemical properties of the composite or composites obtained.

2.1. Component materials used, description, properties

The matrix of composite materials that form the subject of this research is clay. According to [38], loam or clay is a fine-grained sedimentary rock, made up of a mixture of silicates and fragments of quartz, mica, etc. According to [39], the genesis of clays is sedimentary, with small constituent particle sizes, below 0.002 mm and colloidal dimensions, being part of the class of pelitic deposits or loam, according to the nomenclature used in Central Europe. The chemical composition of the clays is characterized, in general, by uniformity, with few differences between the contents of Fe_2O_3, SiO_2, Al_2O_3, MgO, K_2O, Na_2O, TiO_2, CO_2, MnO, SO_3, P_2O_5, H_2O [40].

Clay is a natural, sustainable and simple material, with multiple properties. In the dry state it is crumbly and looks like dust, and in the wet state it is soft and plastic, capable of taking any given shape. After moistening, the clay solidifies and becomes strong and durable.

Clay combined with the right amount of water can form a dough-like mass with plastic properties. Depending on the location of the deposit of this natural raw material, clay is characterized by various quality indicators, thus it is used for various purposes (ceramics, construction material, cement production, technical ceramics, adobe construction, medicine and cosmetology, food clay, and so on). The characteristic properties of clay are [40]:
- high level of plasticity.
- the ability to take any given form.
- refractory properties.
- air and thermal shrinkage capacity.
- excellent agglomeration.
- viscosity of different degrees.
- degree of contraction.
- porosity.
- swelling.
- density.
- waterproof.

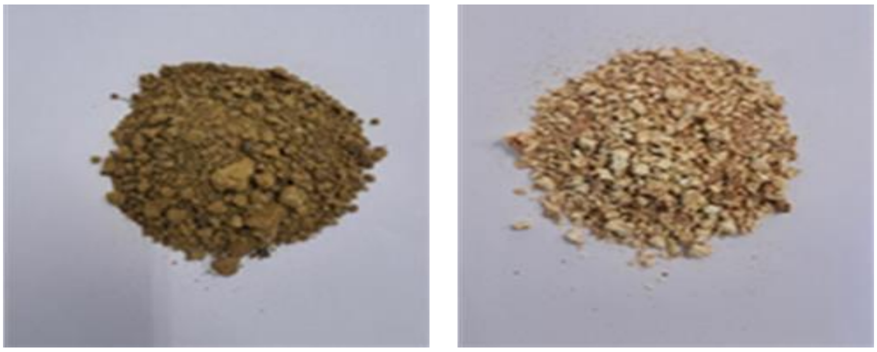
Clay building bricks have high mechanical strength, the ability to harden after firing, giving a material that does not soften in water and is impervious to it. Thanks to the fossil properties, the houses are cool in summer and warm and comfortable in winter.

In carrying out the experiments, the clay from the Horezu area, Vâlcea county (Figure 1), at 475-500 m altitude on the Luncavaț river, at the foot of the North-North East Măguri Slătioara (769 m altitude), at the intersection of the parallel of 45º08' 36" north latitude with the meridian of 23º59'30" east longitude, at 44 km North-North-West from the municipality of Râmincu Vâlcea, in the tourist area of the country known as Nordul Oltenia, which has a tradition in the field of pottery.

Fig. 1. Clay from the Horezu area, Vâlcea county.

The composite materials resulting from the experiments are obtained from clay matrix with agricultural waste insert material (corn cobs), being found in abundance in nature and the behavior in various applications gives these composite materials thus obtained the character of sustainability, both by using as well as through their manufacture.

In this work, the focus is on composite materials (Figure 2) obtained from clay matrices with coccine insert material. The corn cobs were subjected to grinding or chopping processes using an electric knife grinder. Coccine has a volumetric density comparable to spruce wood chips (160-170kg/m^3).



Clay Corn cobs

Fig. 2. Materials used to obtain composites.

2.2. Composite material preparation technology

The technological process of obtaining composite materials involves the use of the following devices: scale, moisture meter, mold, piston, weights, ruler (Figure 3).

Scale Hygrometer Matrix Piston

Fig. 3. Devices used to obtain composite materials.

Composite materials preparation technology:

- the clay is weighed, mixed with water, in order to obtain a homogeneous mixture that has a humidity that allows the formation of a composition that also integrates the insert material in the matrix.
- the obtained composition is inserted into a mold in which it is subjected to pressing in order to eliminate the surplus of water.
- following the compaction of the composite material with the help of a piston actuated by weights, the composite materials (bricks) are obtained within a period.

The bricks made of composite materials resulting from pressing are left to dry for 14 days, in the room at constant temperature (Figure 4) to improve the mechanical properties.

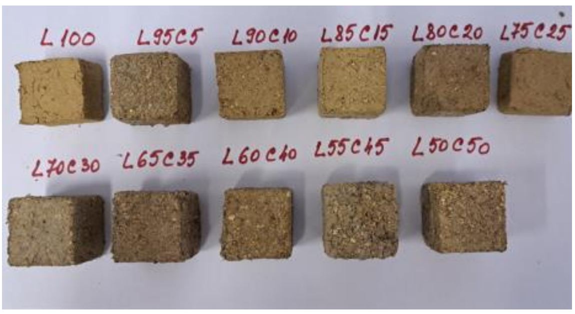


Fig. 4. Composite materials (clay with corn cobs) with different concentrations.

3. RESULTS AND DISCUSSION

The results of a manufacturing process of composite material bricks based on clay matrices are complex and the analysis of the production process requires a large space of experiments and theoretical and experimental analysis. There is a range of results relating to the relationships between the output parameters of the system and the input and control parameters, then a range of relationships that will describe the qualities of the material obtained as a function of the input and control parameters. There are also a series of relationships that must describe the behavior of composite bricks over time, as well as to the action of environmental factors such as excessive humidity, rain, high or low temperatures in relation to the average, the action of rough winds (blizzard with snow or ice), etc.

In this first article, the main relationships between the parameters of the production system will be given, and in future articles, results will be provided about all the qualities of the composite material and its behavior over time and in relation to environmental conditions.

3.1. Definition of the theoretical and experimental framework for the foundation of the material

The composite material whose preparation is described in this article has a clay matrix (with the properties described in 2.1), with coccine inset, having a concentration between 0 and 50 % by mass. The codes of the composite material variants are the following: L100 (control material, made of pure clay), L90C10, L70C30, L50C50, variants whose groups of digits give the numbers representing the concentrations of clay and coke.

Table 1 lists the technological parameters of the manufacturing process of composite materials with clay matrix and insertion of various materials, which characterize the product before and immediately after compression.

Table 1. List of parameters describing the system that models the technological process of manufacturing acomposite with clay matrix and coke insert.

Name	Notation	SI unit of measure
The concentration of the insert in the raw material	c_{mp}	%
Raw material mass	M_{mp}	kg
Raw material mold length	L_{mp}	m
Raw material mold width	l_{mp}	m
The height of the raw material mold	h_{mp}	m
The volume of the raw material mold	V_{mp}	m^3
Raw material mold density	ρ_{mp}	kg/m^3
Raw material mold moisture	u_{mp}	%
Compression time	τ_p	s
Press table (which presses on the piston plate)	m_p	kg
Compression force	F_p	N
The mechanical work of the compression force	L_p	J
The concentration of the insert in the compressed mold	c_f	%
The mass of the pressed material mold	M_f	kg
The height of the pressed mold	h_f	m
The volume of the co-primed mold at the exit from the mold	V_f	m^3
Mold density of pressed material	ρ_f	kg/m^3
The moisture of the pressed material	u_f	%
Compression force	F_c	N

Next, the definitions for the quantities that describe the technological process are given. The volume of the raw material mold can be estimated using the data above:

$$V_{mp} = L_{mp} \cdot l_{mp} \cdot h_{mp} \quad (1)$$

The density of the raw material mold is calculated according to the formula:

$$\rho_{mp} = \frac{M_{mp}}{V_{mp}} \tag{2}$$

Experimentally, the pressing mass, m_p, is known, so that the pressing force is calculated according to the formula (3):

$$F_p = m_p \cdot g \tag{3}$$

where g is the local value of the gravitational acceleration. Since the compression times were approximately timed and the compression height variation was also measured, the values of some synthetic parameters of the compression process can be estimated. An average estimated value of the mechanical work required for compression can be estimated with the formula (4).

$$L_p = F_p \cdot \left(h_{mp} - h_f\right) \tag{4}$$

A measure of the power consumed by the compression of a mold of raw material, can be approximated with the formula (5).

$$P_p = \frac{L_p}{\tau_p} \tag{5}$$

The degree of compression can be calculated as the ratio between the volume of the final product immediately after the compression process is finished and the initial volume of the raw material, as in the formula (6).

$$g_c = \frac{V_f}{V_{mp}} \tag{6}$$

If we assume that the length and width of the mold of material subjected to compression does not change, or even if it changes, the changes can be neglected in relation to the initial and final values, then the degree of compression should also be given by the ratio of the final and initial height of the mold of compressed composite material:

$$g_c = \frac{h_f}{h_{mp}} \tag{7}$$

The compression transformation process of the composite material is not a conservative one, that is, at least the amount of material is not conserved because the material loses water through compression, and the volume obviously changes.

Another synthetic parameter, potentially characterizing the quality of the process, is the power specific to the degree of compression, which is consumed to make the final product:

$$P_{gc} = \frac{P_p}{g_c} \tag{8}$$

In a similar way, the mechanical work specific to the degree of compression is calculated:

$$L_{gc} = \frac{L_p}{g_c} \tag{9}$$

3.2. The variation of the technological parameters with the concentration of the insert

All the physical quantities defined above, in an elementary way, for the analyzed technological process, are dependent on the parameters of the composite material manufactured in the process. The main parameter in relation to which we can do the analysis for now, is the concentration of the insert in the composite, a parameter that can be controlled. Obviously, apart from the concentration of the insert (in this case cocini) there are many parameters that influence the process: density, initial humidity, viscosity, cohesion and adhesion of the composite components, the temperature at which the process takes place, the compression speed and probably others. In this experiment, the dependence of the synthetic parameters of the technological process in the composite made of clay and cocini will be studied. Obviously, the final, qualitative parameters will also depend on these parameters.

The variation in the mechanical work required for compression $L_p = L_p(c_{mp})$ is represented graphically, according to the experimental data, in Figure 5. Mechanical work L_p thus becomes a function of the initial insert concentration in the composite material.

The variation in the power consumed to achieve the compression $P_p = P_p(c_{mp})$ is represented graphically, according to the experimental data, in Figure 6. Power P_p thus becomes a function of the initial insertion concentration in the composite material.

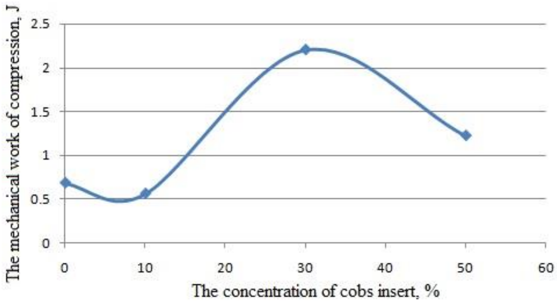


Fig. 5. Variation of the mechanical work of compression.

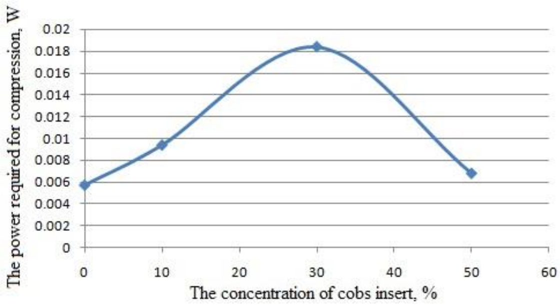


Fig. 6. The variation in average power required for compression.

Compression ratio variation $g_c = g_c(c_{mp})$ is represented graphically, according to the experimental data, in Figure 7. Degree of compression g_c thus becomes a function of the initial insert concentration in the composite material.

The variation of power consumed for compression, specific to the degree of compression $P_{gc} = P_{gc}(c_{mp})$ is represented graphically, according to the experimental data, in Figure 8. The power consumed for compression, specific to the degree of compression P_{gc} thus becomes a function of the initial insertion concentration in the composite material.

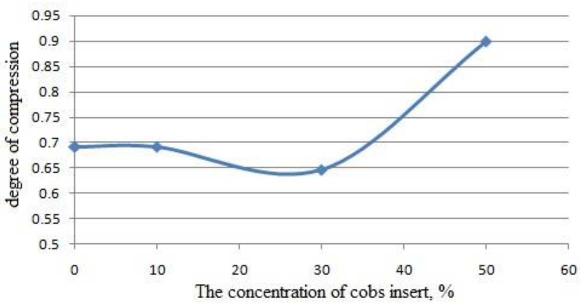


Fig. 7. The variation in average power required for compression.

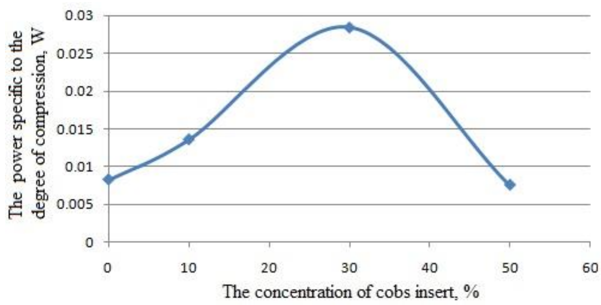


Fig. 8. The average power variation specific to the degree of compression.

The variation of mechanical work specific to the degree of compression $L_{gc} = L_{gc}(c_{mp})$ is represented graphically, according to the experimental data, in Figure 9. The mechanical work specific to the degree of compression L_{gc} thus becomes a function of the initial insertion concentration in the composite material.

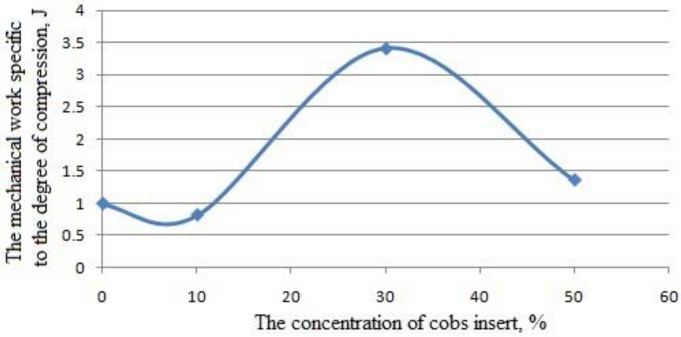


Fig. 9. The variation of mechanical work specific to the degree of compression.

3.3. Study of process parameter variations by interpolation

The variation of the process parameters (technological or process quality) can be done directly on the experimental data, or it can be done by interpolating the basic, experimentally measurable parameters, which thus become functions of the concentration of the insert in the matrix. Subsequently, the synthetic process parameters, which reflect part of the process quality are calculated as functions of concentration using the definitions (4)-(9).

Basic interpolable parameters are the mechanical work required for compression, L_p, compression pressure, P_p, degree of compression, g_c, and the interpolation curves as a function of the material concentration are graphically represented in Figures 10-14.

The polynomial function for the mechanical work required for compression	The relative mean squared error, %
$L_p(c) = 0.747 + 0.019c$	45.921
$L_p(c) = 0.376 + 0.091c - 0.001438c^2$	33.015
$L_p(c) = 0.686 - 0.082c + 0.008281c^2 - 0.0001284c^3$	0.000

Polynomial function for compression pressure	Relative root mean square error, %
$P_p(c) = 0.008868 + 0.0005352c$	48.338
$P_p(c) = 0.004346 + 0.0009371c - 0.00001755c^2$	16.968
$P_p(c) = 0.00571667 + 0.0001689c + 0.00002545c^2 - 0.00000057c^3$	0.000

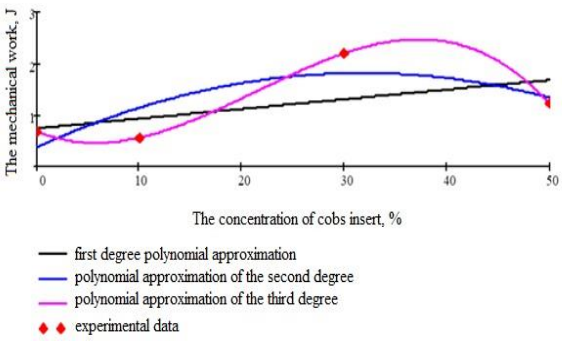


Fig. 10. Interpolation curves for mechanical work required for compression as a function of material concentration.

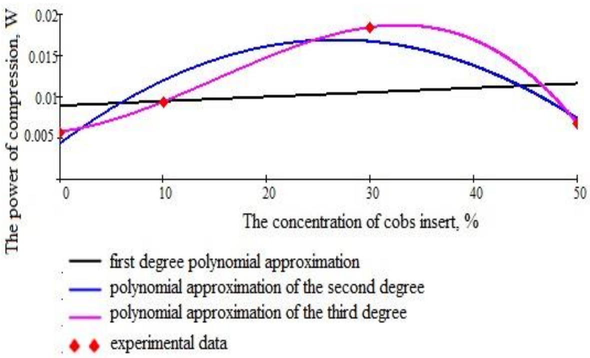


Fig. 11. Interpolation curves for compressive strength as a function of material concentration.

The polynomial function for the degree of compression	Relative root mean square error,%
$g_c(c) = 0.64990774 + 0.00366166c$	9.428
$g_c(c) = 0.71296325 - 0.00865905c + 0.00024475c^2$	3.679
$g_c(c) = 0.69135802 + 0.00343537c - 0.00043311c^2 + 0.00000896c^3$	0.000

The polynomial function for the power specific to the degree of compression	Relative root mean square error, %
$P_{gc}(c) = 0.01346395 + 0.00004425c$	57.736
$P_{gc}(c) = 0.00577754 + 0.00154614c - 0.00002984c^2$	21.482
$P_{gc}(c) = 0.00826875 + 0.00015158c + 0.00004833c^2 - 0.00000103c^3$	0.000

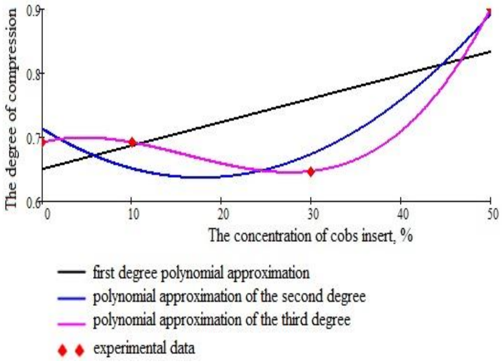


Fig. 12. Interpolation curves for degree of compression as a function of material concentration.

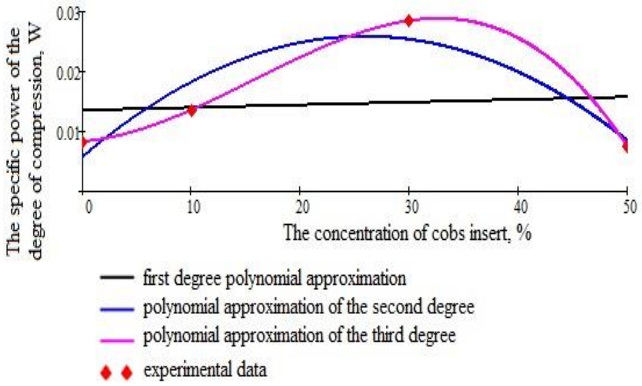


Fig. 13. Interpolation curves for compressive strength as a function of material concentration.

The polynomial function for mechanical work specific to the degree of compression	Relative root mean square error, %
$L_{gc}(c) = 1.17959557 + 0.02067667c$	58.339
$L_{gc}(c) = 0.47491688 + 0.15836718c - 0.00273527c^2$	39.217
$L_{gc}(c) = 0.99225 - 0.13123159c + 0.01349606c^2 - 0.00021448c^3$	0.000

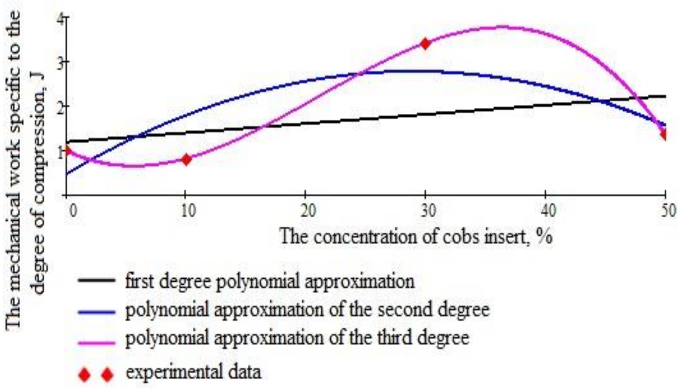


Fig. 14. Interpolation curves for mechanical work specify the degree of compression as a function of material concentration.

3.4. Statistics of technological process variables

In order to establish links between process input and output parameters, it is first important to study correlations. A correlation matrix between some of the input and command parameters and some of the output parameters is given in the Table 2.

4. CONCLUSIONS

The curve in Figure 5 shows that the mechanical work required to compress the raw material has a maximum value at 30 % insert concentration and then decreases as the concentration increases. In the area of low concentrations, the mechanical work of compression seems to have a minimum somewhere in the 7-8 % area.

Table 2. Correlation matrix between input parameters and command and output parameters.

Correlations	Final Mass	Final Height	Final Volume	Final Moisture	Final Density
concentration insert	-0.925	0.981	0.978	0.866	-0.994
weighing table	-0.916	0.931	0.945	0.714	-0.913
press time	-0.712	0.853	0.859	0.769	-0.796
initial height	-0.48	0.754	0.723	0.954	-0.736
initial volume	-0.48	0.754	0.723	0.954	-0.736
initial humidity	-0.917	1	0.999	0.887	-0.994
initial density	0.531	-0.793	-0.764	-0.968	0.776

The curve from Figure 6 shows a maximum point exactly for the same concentration as the curve in Figure 5. The minimum point corresponding to the concentration below 10 % is no longer visible. For results with phenomenological value and/or useful in operation, the increase in experimental resolution must be considered.

The curve in Figure 7 shows that in the maximum point of the mechanical work of compression and the power required for compression, a minimum point of the degree of compression is located. This conclusion is one that still needs to be checked, as it shows that for the highest compression effort, the lowest compression is obtained.

The variation of the average strength specific to the degree of compression, required for compression to obtain the material with clay matrix and coccene insertion with the concentration of coccene, as shown by the curve in Figure 8, is similar to the variation of the curve of power consumed for compression, represented in Figure 6.

The variation curve of the mechanical work specific to the degree of compression varies in relation to the concentration of the insert material, as in Figure 9, variation like that of mechanical work, L_p, from Figure 5.

The results of experimental and theoretical research dedicated to the manufacturing technology of the composite material with clay matrix and coccine insert, show that the systemic description of the technological process is satisfactory.

The main parameters that describe, in the authors' view, the manufacturing process of the composite material were defined. Process quality parameters and composite material quality parameters were also defined. Experimental data were used to obtain relationships impossible to substantiate theoretically. It is found that the proposed technological process leads to materials with properties at least similar to those existing on the market, of course depending on the concentration of the insert.

Considering the results obtained, it is appreciated that it is possible to proceed to the higher stages that include mechanical, thermal and acoustic tests for the composite material with all its variants. After these tests, a more advanced phase will consist in the study of the interaction with the environment, especially the variation of the mechanical, thermal and acoustic properties with the environmental, storage conditions. The behavior of the material under fixed long-term storage conditions will also be interesting.

Essentially the feasibility of the technology and products described in this article is demonstrated. There are a large number of input, output and control parameters that must be varied in the experimental program and that must be entered into the statistical model that will result from data processing. Hard to explain or unexplainable aspects on the graphs presented in this article show that the deterministic model is not suitable for approaching the theoretical construction of the process and therefore statistical modeling should be used for this purpose.

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