ABSTRACT
of the PhD thesis entitled "Research on the use of composite materials in vehicle braking systems"

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CHAPTER 1. THE OPPORTUNITY OF RESEARCH, OBJECTIVES AND EXPERIMENTAL STRATEGIES

The current road safety regulations require the development of new technical solutions for the "vital" systems of the vehicles [1/1].

Many parts of motor vehicles contribute actively or passively to increasing operation performance. *The safety is closely related to the effectiveness of the braking system*, which is one of the most important components of a vehicle [2/2].

Due to the fact that the *automotive industry is a branch of economics that uses products obtained in almost all other industries, being the largest consumer of materials in economy*, the current concerns are directed towards the application of the latest research in the field of modern materials and technologies in all fields involved in the manufacture of motor vehicles [3/4].

The scope of the PhD thesis is the approach of 3 research directions:

- Obtaining in laboratory of organic composite materials according to own recipes, designed to make brake pads for small vehicles and medium performances;
- Technological analysis and optimization of the qualitative properties of the friction materials produced;
- Characterization and testing of organic composite materials produced according to own optimized recipes, according to the standards in force.

The research has an applicative character, the obtained results having practical applicability in the field of motor vehicle engineering.

The PhD thesis has a multidisciplinary character and represents a contribution in the field of materials engineering intended for the braking systems of the road vehicles. The proposed and carried out researches and experiments aimed to obtain, characterize and test some organic composite materials, designed to make brake pads for the braking systems of small cars with medium performances.

CHAPTER 2. ANALYSIS OF VEHICLE BRAKING SYSTEMS COMPONENTS

This chapter contains an overview of the main components of the vehicle braking systems, highlighting their operating principles, analyzing the role of brake discs and pads, presenting the requirements for these co-functional elements for avoiding safety hazards, as well as the importance of using composite materials in the production of brake pads.

The brake discs and pads are the most demanded components for the safety of the braking system, which work together, in their realization being imposed some requirements that must be met by each of them. Subject to these requirements, the safety risks, loss of comfort and reduction in the durability of this co-functional assembly can be avoided by [4/12]:

- Use of appropriate materials to meet the lot of requirements imposed on both co-functional components;
- Use of modern manufacturing technologies;
- Correct maintenance of the brake disc-pad assembly.

In this context, the current research directions in the automotive industry are directed to the materials and manufacturing technologies of their components.

CHAPTER 3. THE CURRENT STAGE OF RESEARCH ON THE MATERIALS USED IN THE MANUFACTURE OF BRAKE DISCS AND PADS

This chapter includes a bibliographic synthesis on the evolution of materials used in the manufacture of brake discs and pads.

The vehicle brake discs are made of cast iron, being the most technologically and economically efficient material that meets all the properties required by this braking system component [5/13]. Under actual operating conditions, the brake disc is exposed to a thermo-mechanical fatigue stress and, therefore, the materials used to make the brake discs must have high heat capacity and thermal diffusivity, low density, must ensure a good coefficient of friction, stable mechanical properties at high temperatures in wet environments, high wear resistance, corrosion resistance, friction stability, low weight, low noise, operating durability and, last but not least, cost price as low as possible [5/13]. Over time, the properties of the cast iron used to make brake discs have been improved by adding alloying elements, such as: manganese, vanadium, molybdenum, chrome, copper, etc. [6/19].

In the iron casting process for making brake discs, both the chemical composition and the factors influencing the cooling process must be rigorously controlled. In this regard, the main parameter is the cooling rate which depends on the wall thickness of the part, the thermo-physical properties of the casting mould, the characteristics of the casting technology (temperature, casting speed, casting mould temperature) [7/20].

The most used cast iron type used in the manufacture of brake discs is the grey cast iron with lamellar, nodular and vermicular graphite, and the microstructures that provide the best performance in the use of brake discs are the ferrite-pearlite and pearlite ones. [8/21].

The brake discs can have a direct effect on the durability of the brake pads, which are an important component of the vehicle braking system. The brake pads may be of different types, their choice depending on the type of vehicle, the type of engine, and the general characteristics required by the standards in force [9/45].

The type of brake pads depends on the material from which they are made, and can be: organic, semi-metallic, metallic and ceramic [9/45].

The composite materials do not exist in nature, but they are synthetically produced to meet well-defined requirements. By means of a qualitatively and quantitatively appropriate choice of the composite materials, we can obtain composite materials with superior properties compared to the traditional materials [1/1], [2/2]. In the design of a composite material, it is intended to obtain high resistance and high rigidity relative to the unit of weight, the parameters being expressed by two characteristics: the specific strength and the specific modulus [10/46].

The main categories of materials used in the composition of organic brake pads are: abrasive materials, friction modifiers, fillers and binders [11/48].

The friction coefficients for brake disc-pad friction couplings are 0.3 - 0.7, but the brake pads used in the urban traffic have an average friction coefficient of 0.25 - 0.35 [12/63].

The main defects of the brake discs are: brake shocks, operating noise, cracks and scratches, and the main defects of the brake pads are: detachment of the friction material, use-related defects, faulty installation, and damage caused by environmental influences. Obtaining and preserving the braking qualities of a motor vehicle can be achieved by the use of some brake components that have no defects [4/12].

In conclusion, thanks to their qualities, the composite materials are superior to the classical materials. In the automotive industry, the use of these materials increases the strength of the components, while reducing their weight, which leads to lower fuel consumption and increased performance.

CHAPTER 4. TECHNOLOGICAL EXPERIMENTS AND ANALYZES ON THE OBTAINING AND CHARACTERIZATION OF COMPOSITE MATERIALS

Within this chapter, several recipes of composite materials have been developed, analyzed, optimized and characterized, for making brake pads for small vehicles and medium performances.

In the first stage of the experimental research programme, two moulds were designed and made, in order to produce the samples necessary to determine the physical, mechanical, and tribological properties of the composite materials. Also, 20 recipes have been designed and materialised, and the sintering technology has been developed step by step, on the basis of the results of technological analyzes afferent to each recipe. We selected a number of 8 recipes to make disk-shaped and cylindrical samples (pins), from which specimens were taken to determine the physical, mechanical, and tribological properties.

In the second stage of the experimental research programme, the obtained materials have been characterised by experimental determination of the physical and mechanical properties, and the initially-established recipes have been optimised by statistical-mathematical calculation.

In the third stage, the composite materials were produced according to the optimized recipes for which the physical and mechanical properties have been determined experimentally, being also carried out the surface morphology study.

In order to establish the recipes presented in this chapter, we used a mixture of raw materials made of aluminium, graphite, zirconium oxide, silicon carbide, titanium oxide, aluminium oxide, sulphur, phenolic resin (of Râşnov type) and coconut fibre. Their choice was made with regard to the purpose of the brake pads (for small vehicles and average performance), according to the literature on the composition of organic brake pads. The laboratory production of organic composite materials for brake pads is based on the powder metallurgy [13/64].

Eight samples were selected from the existing 20, considered to be appropriate in terms of compaction, integrity, elasticity and appearance of the sample, which were used in the following experiments. The 8 recipes can be grouped into two families of composite materials, due to the similar composition of their components. Within the same family of composite materials, about 75% of constituents were maintained in the same proportion, and the proportion of aluminium and coconut fibre have been modified by ± 5g (5%). The difference between the two families of composites consists in the fact that the first family has titanium oxide, and the second family has aluminium oxide, both components being found in identical proportions. Cylindrical samples were made from each recipe, from which specimens were taken to determine the hardness, density, porosity, and surface morphology analysis, as well as for performing mechanical tests. The conclusions reached in determining the physical and mechanical properties of the composite materials are as follows [14/65]:

- The density values for the two families of composite materials produced are relatively low compared to the metal densities;
- The specimens produced based on the recipes that do not include coconut fibre have the highest density, due to the fact that these recipes contain the largest amount of metal;
- The lowest value of porosity was obtained for the samples containing 10% coconut fibre;
- It can be noticed a tendency of compressive strength increase with the increase of the coconut fibre proportion;
- By comparing the obtained results with those found in the literature, it is noted that the values obtained for the compressive breaking force, breaking stress and longitudinal elastic modulus are relatively low;
- For some samples, the tear did not occur at the maximum force of the test equipment, i.e. 5 KN, so these samples have resistance reserves, requiring the tests to be repeated using testing equipment which enables a higher breaking force.

Taking into account the obtained results, the recipe components could be optimized.

Thus, 15 recipes were created, in which the percentage of aluminium varied from 6 to 25 (%), and the percentage of coconut varied from 0 to 19 (%).

From each recipe, disk-shaped samples were made, for which the compression resistance has been determined.

The results were processed in Excel and Matlab, in order to obtain correlations between the compressive strength and the proportion of components in the recipe, on the basis of which the optimal recipes for the production of composite materials to be found.

Fig. 1/4.37 presents the compressive strength versus the amount of aluminium and coconut fibre for the F1 family of composite materials, and Fig. 2/2.48 for the F2 family.

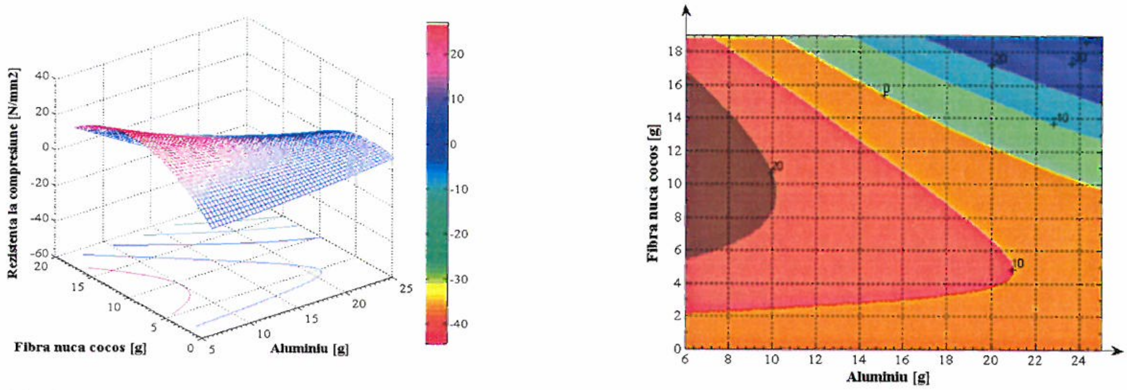


Fig.1/4.37 Variation of compressive strength versus the amount of aluminium and coconut fibre for the F1 family of composite materials

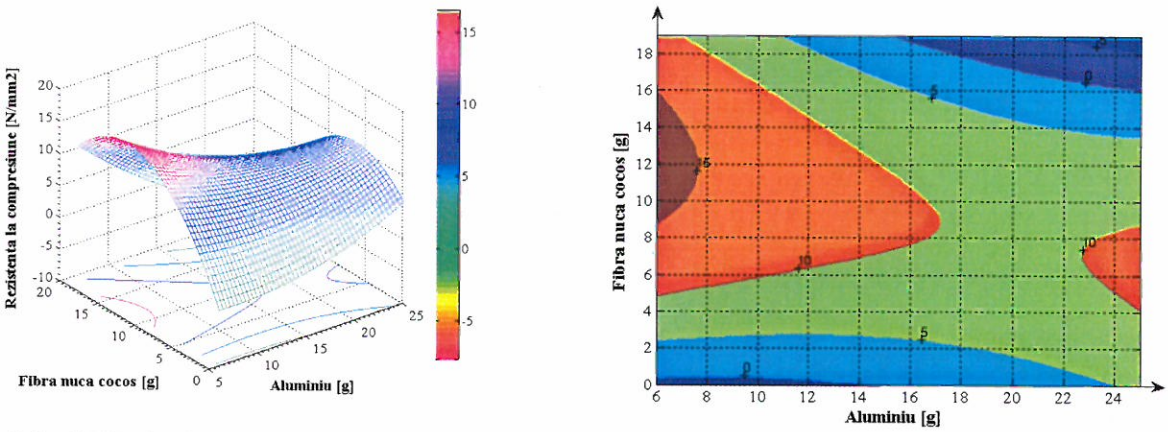


Fig.2/2.48 Variation of compressive strength versus the amount of aluminium and coconut fibre for the F2 family of composite materials

The technological domain for the aluminium content ranges from 10g to 20g (10-20%), and for the coconut fibre from 6g to 14g, providing the best mechanical compression properties for the composite materials produced.

For the complete characterization of the two families of composite materials, the study of sample surface morphology was performed through SEM-type electron microscopy and EDX analysis. The conclusions drawn from these analyzes are:

- The particle distribution in the friction material differs from one sample to another;
- The material particles have different sizes and are unevenly dispersed in the matrix;
- The samples do not have the homogeneity of the constituents;
- The EDX analysis highlights the similarity of the elements of composite materials produced with the original ingredients introduced in the recipe.

The organic composite materials are heterogeneous materials made up of several components with various properties, and hence the selection of constituents and their percentage in recipes will significantly affect the behaviour in operation. For this reason, in order to obtain superior physical and mechanical properties, we made changes in the initially produced recipes and improved the sintering technology.

Considering the technological domain obtained for the recipe components as a result of the optimization studies, four recipes have been used to produce composite materials, whose chemical composition is shown in the Tables 1/4.12 and 2/4.13, two for each family F1 and F2, respectively.

Table 1/ 4.12

Optimized chemical composition of composite materials belonging to the F1 family

Samples	Aluminium [%]	Grafite [%]	Zirconia oxide [%]	Silicon carbide [%]	Titan oxide [%]	Phenolic resin [%]	Hexametyltetramine [%]	Coconut fibre [%]
C1	20	5	2	11	11	40	6	5
C2	15	5	2	11	11	40	6	10

Table 2/ 4.13

Optimized chemical composition of composite materials belonging to the F2 family

Samples	Aluminium [%]	Grafite [%]	Zirconia oxide [%]	Silicon carbide [%]	Aluminium oxide [%]	Phenolic resin [%]	Hexametyltetramine [%]	Coconut fibre [%]
C3	20	5	2	11	11	40	6	5
C4	15	5	2	11	11	40	6	10

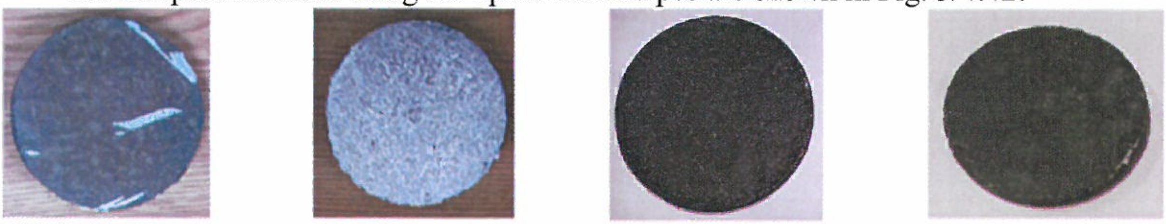
The parameters of the sintering technology have been established as a result of several experimental tests, enabling us to carry out critical analyzes to successively improve the technology. The sintering technology parameters are shown in Table 3.

Table3

Parameters of the sintering technology

Heating temperature [^{0}C]	Maintenance time in the oven [min]	Pressing force [KN]	Heating - cooling regimen			
			In oven		In air	
			Temperature [^{0}C]	Time [min]	Temperature [^{0}C]	Time [min]
200	60	20	100	480	25	2880

The samples obtained using the optimized recipes are shown in Fig. 3/4.42.



C1 (5 % cocos) C2 (10 % cocos) C3(5 % cocos) C4 (10 % cocos)

Familia F1 (TiO_2) **Familia F2 (Al_2O_3)**

Fig.3/4.42 Samples obtained after optimized recipes

Analyzing the samples obtained when using the four recipes optimized in terms of compactness, integrity, elasticity and appearance at the extraction from the mould, the following conclusions could be drawn:

- All samples preserved their integrity when were extracted from the mould;
- The homogenization and solidification of the constituents show that the order of the operations for obtaining the composite material is good;
- All samples have a smooth surface, a completely circular geometric shape, appropriate hardness and consistency;
- The heating - maintaining - cooling regimen is appropriate, and the hot and cold compression force applied led to an efficient compaction of the material;
- All samples are considered successful and can be used for specimen sampling, required to describe the composite materials produced based on the optimized recipes.

These samples were analyzed in terms of physical and mechanical properties, and comparative graphs for density, porosity in oil and water, hardness, and compressive strength have been drawn. The conclusions are:

- The density of organic composite materials produced based on optimized recipes increases with the increase in the amount of coconut fibre;
- The decrease of the level of porosity, both in water and in oil, as a result of the increase in the bond between the binder and the filler, was determined by the proper homogenization of the recipe components;
- The hardness, compressive strength and longitudinal modulus of elasticity increase with the increase in the amount of coconut fibre;
- The factors influencing the reach of superior physical and mechanical properties by the composite materials are: the type of components chosen for the recipes, optimization of the proportion of components, and the parameters of the sintering technology;

Next, we studied the surface morphology of the composite materials, using the SEM microscopy and EDX analysis. In the SEM images, we can see that:

- The material particles have different sizes, but are uniformly dispersed in the matrix;
- The carbon can be found in all structures and dominates the other constituents; it was homogeneously distributed among the recipe constituents.
- The aluminium is found in all samples;
- A good dispersion of the binder and coconut fibre in the aluminium matrix could be noted, making a suitable interface between the filler material and the aluminium matrix.

The EDX analysis showed a relatively good combination of the constituents, without changes in their proportion.

The conclusions resulting from the laboratory characterization of the composite materials produced according to own recipes are:

- The structural integrity of composite materials provides superior physical and mechanical properties;
- The factors influencing the physical and mechanical properties of the composite materials are: type of the components chosen in recipes, optimisation of the proportion of components, parameters of the sintering technology.

CHAPTER 5. TRIBOLOGICAL BEHAVIOUR OF THE COMPOSITE MATERIALS USED TO MAKE BRAKE PADS

This chapter presents the tribological behaviour of the composite materials produced in compliance with the optimized recipes. The experimental research strategy included the determination of the functional characteristics, the friction coefficient and the temperature in the friction coupler area. In this respect, experimental laboratory determinations were carried out on the wear of the samples of composite materials produced according to optimized own recipes, using standardized test methods. The purpose of the tribological research carried out on composite material samples was the influence of some material factors (concentration and nature of the constituents) and some parameters of the working regime (compressive force, sliding speed, testing time, friction regime) on the tribological properties of the tested samples. The knowledge of these elements provides the possibility of pertinent assessments on the durability in operation of the composite materials used for the manufacture of brake pads.

In order to carry out this study, we made disk-shaped and pin-shaped specimens of cast iron, intended to be installed in wear systems which enable studying the friction and wear behaviour of the composite materials produced. In this respect, two cast iron heats were made, which were analyzed in terms of chemical composition, hardness and metallographic structures.

Analyzing the chemical compositions of the two cast iron heats comparatively with the chemical composition of the brake discs stipulated by ASTM A159, it is noted that:

- The cast iron made in the first heat corresponds to the grade G 2500;
- The cast iron made in the second heat corresponds to the grade G 3000.

Thus, the first heat has been chosen, being intended for the production of brake discs for motor vehicles. Disk-shaped and pin-shaped specimens were made from this cast iron heat, in accordance with the testing equipment, for finding the functional characteristics of the composite materials. Therefore, we studied:

- The influence of the mass and dimensions on the wear parameters;
- The influence of speed and compressive force on the wear parameters.

The tribological equipment for testing composite materials is based on the dry friction regime, and the testing method is *pin-on-disk*. The main parameters used in tribological experiments are [15/66]:

- Sliding speed: v = 3.92 m/s and 4.71 m/s;
- Testing radius: R = 25 mm;
- Distance (length) covered: L = 2000 m;
- Testing time: t = 8.5 min.

The wear intensity and wear durability were determined by testing. The highest wear intensity was obtained for the "C1" composite and the lowest for the "C4" composite. Also, for both families of composite materials the highest relative wear resistance was obtained for the composites containing 10% coconut fibre (C2 and C4). The lowest mass wear was obtained for the sample C2 (F1 family of composite materials), followed by C4 (F2 family of composite materials), both of them being made using 10% organic fibre. The conclusions are:

- The "C2" sample has the highest wear resistance, being followed by C4, both of them containing 10% organic fibre;
- The wear resistance is also satisfactory for C1 and C3 samples, which contain 5% organic fibre;
- The wear of the cast iron disc is much lower than the wear of the composite materials, which shows that the wear resistance does not depend too much on the amount of metal part, which is very important for the practical situation because the brake pads can be more easily replaced than the discs.

The studied materials were further tested under dry friction conditions, using three compressive forces: F = 5 N, F = 10 N and F = 15 N, and two sliding speeds: 3.92 m/s and 4.71 m/s, noting the evolution of the wear rate. The conclusions are:

- The linear wear rate decreases as the sliding speed increases;
- The linear wear rate decreases as the compressive force increases;
- The samples C2 and C4 have the best wearing behaviour, being made with 10% organic fibre.

The evolution of the thermal field in the contact area of the friction couplers was also analysed in this chapter. The thermal field was analyzed using a thermographic camera. The captured images can provide information on the temperature evolution in the pin-disc contact point, as well as the contact print temperature.

The experiments were carried out applying three different load forces: 5N, 10N and 15N, the distance to be covered being 500 m - 2000 m. The conclusions are:

- For all four pins (C1-C4), the temperature in the contact area rises fast in the first testing stage, until the distance of 500 m is reached;
- In the range of 500 - 1500 m, the temperature rises less and we can say that the temperature stabilization process begins in this range of covered length;
- Most of the heat dissipates in the pin, which in the real friction coupler represents the brake pad;
- The lower is the temperature in the contact area, the greater is the heat dissipation; therefore, the degradation of organic components in composite materials is lower;
- The C2 and C4 pins have the best behaviour, both of them containing 10% organic fibre.

Then, we studied the influence of some parameters of the working regime (e.g. compressive force, rotative speed, testing time) on the tribological properties of the tested samples. In this respect, the evolution of the friction coefficient has been studied using a TR-20 tribometer, whose principle of operation is based on the "pin-on-disk" method. The pin of the equipment consists of a 6-mm diameter steel ball, and the friction regime applied for testing is "dry friction".

In the experiments, the composite material specimens were made in a parallelepiped shape and weighted using a digital scale with an accuracy of ± 0.01 mg. Three successive weights were made and their average was taken into account. The wear was determined for each sample, by mass loss between the value measured initially and the value measured at the end of the test.

The testing parameters used in the experiment are [16/71]:

- Diameter of the wear print: D = 15 mm;
- Rotative speed: n = 150 rot/min;
- Testing time: t = 5 h;
- Testing distance: d = 2200 m.

The experiments were carried out for 3 loading forces (5 N, 10 N and 15 N, recording the evolution of friction coefficients. The conclusions are:

- For all compressive forces, the friction coefficient becomes stable after a relatively long period of time from the beginning of the experiments, this being confirmed by the following facts: the temperature in the contact area of the friction couplings increases in the first 1500 m of the distance covered, and only then it begins to decrease; the temperature decrease indicates a higher heat dissipation and a stable value of the friction coefficient.

The influence of the compressive force on the friction coefficient was also studied in this chapter, as well as the influence of the compressive force on the sample wear.

- The friction coefficients decrease with increased compressive force;
- The wear decreases with increased compressive force.

CHAPTER 6. FINAL CONCLUSIONS. ORIGINAL CONTRIBUTIONS. FUTURE RESEARCH DIRECTIONS

The conclusions reached at the end of this paper are:

- The braking system qualities can be assured by using materials suitable for the manufacture of its components, using modern manufacturing technologies and a proper maintenance of the brake-disc assembly;
- A compromise between safety, comfort and economic efficiency must always be found in the manufacture of the brake pads;
- When the composite materials are made in laboratory, the factors that influence the homogeneous mixture are the mixing speed and the amount of phenolic resin, as well as the order of mixing the components;

- The improvement of the composite materials properties can be achieved by making changes in the concentration of the components in the recipes, as well as by changing the parameters of the sintering technology;
- The factors influencing the reach of superior physical and mechanical properties by the composite materials produced are the type of components chosen in the recipes, the optimization of the proportion of the components and the parameters of the sintering technology;
- Because of knowing some wear parameters, the temperature in the friction coupler contact area and the evolution of the friction coefficient under various compressive forces, we could assess the operation durability of the composite materials;
- For all composite materials tested, the linear wear rate decreases with increased slipping speed and increases with increased compressive force;
- For all composite materials tested, the friction coefficients decrease with the increase of the compressive force and become stable after a certain period of operation;
- The friction and wear properties of the friction materials depend on the compressive force, slipping speed, temperature in the contact area, anatomical structure of the friction material, travel duration and length;
- The samples C2 and C4 have the best tribological properties, both of them containing 10% organic fibre; we specify that C2 is part of the first family of composite materials produced (which has titanium oxide in the recipe), and C4 is part of the second family (which has aluminium oxide in the recipe);
- The average friction coefficients obtained for the samples C2 and C4 determine these materials to be included in the category of materials suitable for making brake pads for small vehicles and medium performance, designed for urban traffic;
- The results obtained in this paper qualify the coconut fibre as a filler material for the composite materials designed to make brake pads for small vehicles and medium performance.
- The inconvenience of producing the composite materials analyzed in this PhD thesis is the relatively high price of the raw material, the coconut being an exotic fruit.

The original contributions of this paper are:

- Study of the literature on the components of the braking system, the factors influencing the operation of the brake-disc assembly, the materials used to make brake discs and pads;
- Designing two moulds for making disk-shaped and cylindrical (pine) samples from the developed composite materials and improving the moulding solution for the production of disk-shaped samples;
- Determining the composition of the 20 recipes for the composite materials produced in the laboratory;
- Establishing the succession of sintering process operations for obtaining composite materials, determination of their physical and mechanical properties, and critical analysis of the obtained results;
- Making 15 samples with various concentrations of aluminium (6-25%) and coconut fibre (0-19%) and determination of mechanical resistance to compression in order to obtain the required data to optimize the recipes by statistical-mathematical calculation;
- Data processing in Excel and Matlab computing programs, in order to establish the technological domain for the composition of recipes;
- Determination of the technological domain for the concentrations of aluminium and coconut fibre, which led to the improvement of the physical and mechanical properties;
- Determination, formulation and completion of the optimized recipes;
- Modification of the composite material sintering technology in accordance with the optimized recipes, and making them in the laboratory;

- Determination of the physical and mechanical properties and the study of surface morphology;
- Comparative analysis of the physical and mechanical properties of the composite materials produced using the two variants of recipes (initial and optimized);
- Making two heats of cast iron in the laboratory to determine the tribological properties of the composite materials (in the laboratory stage), followed by their technological analysis in accordance with the standards in force;
- Experimental determinations regarding the evolution of wear parameters, in order to assess the tribological behaviour of the materials produced;
- Study of the influence of the mass and dimensional properties of the specimens, made from composite materials produced using optimized recipes, over some wear parameters;
- Study of the influence of the speed and compressive force over the wear parameters;
- Analysis of the thermal field evolution in the contact area of the friction couplers (cast iron - composite material and composite material - cast iron);
- Analysis of the wear and friction coefficient evolution under the action of various load forces.

As any research that has been developed on a given topic, it can not be said that it is finalized at some point, because the evolution in the field of brake pads opens up opportunities continuously for development and deepening, by resuming or imposing new operation techniques.

The future research directions are:

- Tribological testing of composite materials at sliding speeds and loading forces higher than those used in this study;
- Highlighting the wear processes occurring in the superficial layer of the analyzed samples;
- Study of the thermal fatigue phenomenon occurring during operation at the contact between the brake discs and pads;
- Brake performance testing according to ECE Regulation 90;
- Assessing the durability, lifecycle and occurrence of defects;
- Development of a brake pad manufacturing system at industrial scale;
- Use of other economically advantageous organic fibres, or a combination of fibres (flax, rapeseed, reed) in the production of friction materials.

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