1. Target/Research hypothesis/Research plan

In general, the project aims to establish the basis of theoretical and experimental methods for 3D structuring of graphene flakes (G) previously received in the way of electrochemical exfoliation of high purity graphite. **The new knowledge on the principles of 3D structuring of graphene will open new practical applications for such materials as adsorbents, catalysts and electrode material in electric power devices like fuel cells and batteries.**

Anodic and cathodic electro-exfoliation will be applied as well as different electrolyte solutions based on water. New knowledge will be established during the study of the chemical properties of exfoliated G in conjunction with the composition of electrolytes containing substances which split off:

- anions (anodic exfoliation), which after the oxidation at anode will react with exfoliated G flakes generating a number of products in the form of functional groups of different nature and bonded to the exfoliated flakes; at first the following electrolyte additives will be tested: organic compounds (carboxylic acids, amino acids) and inorganic compounds (sulphates, hydroxides, phosphates, nitrates of Na and/or K),
- cations (cathodic exfoliation), which after the reduction at cathode will produce chemical changes of the flakes in the form of the metal derivatives (nano-sized) and the functional groups of various type bonded to the exfoliated flakes; at first the following electrolyte additives will be tested: organic compounds (phenols, amines, amino acids) and inorganic ones (chlorides/acetates/nitrates of La, Mn, Ti, Ce or cations of these metals in a complexed form).

The process of electro-exfoliation will aided by the presence of anionic/cationic/non-ionic surfactants. At this stage, a separator/template it will be introduced to permanently separate the flakes of split G and its derivatives. The separators/templates will be used as a water-insoluble powder with grain diameter of 10-50 nanometer range. Other separators/templates will be *in-situ* precipitated as nano-crystallites due to crystallization upon the progressive removal of water.

The functional groups formed during the anodic and cathodic exfoliation G will be tested as useful for the fixation of the 3D structured G (with the entered separator/template) by the reaction of the functional groups with selected bifunctional crosslinking reagents (for example: dialcohols, diamines, amino acids, polyvinyl alcohol, chitosan, etc.). In the case of inefficient crosslinking i.e. chemical fixation of the flakes into a 3D structure, physical binders will be applied (already verified experimentally - own research unpublished). The fixation effect will be achieved by the admission of water-mixable polymers like polyvinyl acetate or water-mixable monomers like furfuryl alcohol followed by its polymerization.

The removal of separator/template is the final step in the procedure the 3D structuring of G the action of water and/or acids, for example HCl. In this stage the opening of pores will happen giving the material properties typical for an adsorbent/catalyst: enhanced surface area (the expected value is greater than 500 m^2/g) and pore structure in the mesopore range (expected pore diameter 10-50 nm).
For comparative purposes, it is planned to conduct a series of experiments with the exfoliation of graphite plates G in the presence of dipolar aprotic solvents.

At each stage of the study a wide range of instrumental methods will be applied for the characterization of the properties of materials obtained on the basis of G. It is intended to carry out the study:

- the physical structure (specific surface area, pore size distribution by gas adsorption methods; crystalline substances identification by XRD, HRTEM and XPS),
- the chemical composition (FT-IR, TPD-TPR, DTA-TG-DTG, SEM-EDX, HRTEM-EELS, XPS, Raman spectroscopy),
- the catalytic activity and electrode performance (microreactor technique coupled with gas chromatography mass spectrometer GC and MS and voltammetry).