Physical and functional properties of the materials

Abstract

This thesis of habilitation in Physics contains 9 chapters about some scientific achievements of the candidate, after obtaining the title of doctor in Physics.

The first two chapters focus on calcium fructoborate and boric acid with hexagonal structure. The next two chapters present the effect of the processing of AgCu/Cu2O nanocomposite by mechanical alloying, on the physical properties and the structure and thermal behavior of nanostructured AgCu system at repeated melting. Chapter 5 is a research about the condition monitoring of power transformers oil using thermal analysis and other physical techniques. In Chapter 6 it establishes the degree of polymerization of a light-cured methacrylate-based composite material used in dentistry. The last three chapters study the physical properties of azomonoethers, which have properties of liquid crystals, corrosion inhibitors or antimicrobial activity.

In this summary I will refer briefly, only the findings of this research, that highlight practical importance of the materials studied.

Thermal analysis of calcium froctoborate (FruiteX B), boric acid and calcium carbonate showed that there are no similarities between their thermoanalytical curves. Only with fructose, similarities appear, but shifted to higher temperatures as a result of the bonding influences in the calcium fructoborate complex. Mass loss is of 8.5 % up to 152 ºC. The correct molecular formula of FruiteX B has been identified as $Ca[(C_6H_{10}O_6)_2B]_2 \cdot 4H_2O$, which contains two times more boron than reported in other researches. XRD results show a weak crystallinity of calcium fructoborate and confirmed the thermal analysis results when no evidence of fructose, boric acid and calcium carbonate were found. FruiteX B comes therefore as the calcium fructoborate found in natural products.

A special preparation procedure has leaded to the change of the crystallization system of boric acid from the triclinic to hexagonal, as confirmed by XDR analysis. The XRD diffraction maxima of HBA powder permitted the calculation of lattice parameters (a = b = 20.4869 Å and c = 12.1506 Å), and of the mean value of the particle dimensions (of about 19.77 nm). By optical microscopy, we observed the evident hexagonal prism structure of monocrystals grown from water solution of HBA, as well as the strong anisotropy of the exotic HBA polycrystalline structures. The dielectric properties of HBA & H2O solutions proved polar character of the compound. Thermal analysis of HBA, performed under a heating rate of 10 K/min, has shown a good stability till 101ºC, and then decomposition into three stages, all endothermic, but having a different kinetics compared to TBA. The calculations give an energy of 4.40 eV for the decomposition of a TBA molecule, while for the decomposition of a HBA molecule is needed an energy of 4.68 eV.

In order to obtain further insight into physical mechanisms explaining the properties of the AgCu composite nanoparticles reinforced by in-situ Cu_2O synthesis, in the present work combined XRD, XPS, TG/DSC, drop calorimetry and TMA experiments were performed and the composite data were analyzed to make clear the relationship between processing parameters, the particle size distribution and chemical composition of the synthesized materials, and their thermal behavior.

This study concerns the influence of the wet mechanical alloying applied to Ag-28 % Cu micronic powders mixture on the structural development along 80 hours processing respectively the thermal behavior during the first and the second melting. From this point of view, the performances are justified by the AgCu particles structure that changed from elemental micronic powder particles mixture into composite nanoparticles. The structural modification is due to the wet mechanical alloying conditions providing in-situ synthesis of Cu_2O as reinforcement component of the bimetallic AgCu nanostructured matrix. The experimental data on the desorbtion energy, in the

range of 1.50 – 1.99 eV/molecule, confirm the chemosorbtive reaction of argon which intensity depends on the nanoparticles specific surface.

The transformer insulating oil provides valuable information on the actual condition of the transformer. These oil concerning data make it possible to anticipate potential failures (defects) in the transformer and thus to achieve a proper maintenance and a well-developed plan for reconditioning or replacing the oil. This chapter presents the results of experiments performed on some samples of mineral oil that were taken over four and a half years of monitoring, in three stages, starting in 2009. Analysis results (across the entire monitoring period of oil) concerning DGA, FTIR, and TA and determination of electrical, physical, and chemical properties of oil allowed to conclude that oil showed high thermal stability, in terms of mass loss, in the temperature range of interest during transformer operating, for the entire monitoring period.

Spectral analysis of the commercial restorative composite material (Pekalite) shows that increasing the curing time ensures a higher degree of polymerization. TA of the composite samples light cured for different times shows that with increasing the exposure time, the thermal stability of the samples increases. Growth of photo-polymerization time from 5 to 60 s produces an improvement in the mechanical strength of the composite material from 143 to 248 MPa.

A new azomonoetheric dye (CODA) with liquid crystalline properties has been synthesized. Detailed ^{1}H- and ^{13}C- NMR study (400 MHz) of CODA compound using mono and bidimensional NMR spectra was reported. The results are in agreement with the considered molecular structure. By XDR analysis, the main diffraction maxima of the CODA compound have been identified since it has exhibited a good crystallinity and the size of crystallite was established. CODA compound has been indexed as crystallizing in orthorhombic symmetry. The thermophysical processes were monitored by heating–cooling cycles, but the formation of liquid crystal phases were observed only for small values of the cooling rates, when they would separate.

A sulfonated azo compound named 4-(phenyldiazenyl) phenyl benzene sulfonate (PPB) has been synthesized, and its structure has been assigned using UV–Vis spectrometry, FTIR, MS, and ^{1}H-NMR. The thermal analysis, galvanostatic technique and antimicrobial activity of PPB were carried out.

This study deals with the non-isothermal kinetics of three liquid crystals from the series of new aromatic azomonoethers. The evolved gases were analyzed by FTIR spectroscopy, indicating oxidative decomposition for two of them and evaporation for the third. MAPLE technique was used to obtain thin films of 4CN type liquid crystal and to surpass the encountered difficulties of PLD concept and procedure. FTIR spectroscopy results indicate the preservation of the initial organic compound on each substrate.