MAGNETIC FLUIDS AND THEIR TECHNICAL AND BIOMEDICAL APPLICATIONS

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ABSTRACT

Magnetic fluids are stable colloidal systems of fine single-domain magnetic particles (Fe_3O_4 or γ-Fe_2O_3) that are suspended in liquid carrier such as water, mineral oil, damping oil, paraffin, kerosene and so on. The typical size of the magnetic particles is at about 10nm in diameter with magnetic moment of $10^{-4}\mu_B$. The properties of magnetic fluids are well controlled by external magnetic field. In this review we want to discuss briefly history of magnetic fluids, their basic properties, technical applications such as sealing, floatation, loudspeaker, inclinometer, power transformer and biomedical applications such as magnetic drug targeting, magnetic resonance imaging, biomagnetic separation and hypethermia. The main results of the technical and biomedical applications research in our Institute will be presented.

KEYWORDS: magnetic fluid, fine magnetic particles, technical and biomedical applications

1. INTRODUCTION

Magnetic fluids have existed in one form or another for over 200 years. Although intensive research on magnetic fluids did not start until the 1960, the preparation of water based magnetic fluids had already been described in 1938 by Elmore [1]. The first ferrofluids were primarily used as a means to study magnetic domain structure in solids. The modern era of ferrofluids manufacture begins when ferrofluids were made using colloidally stable particles using the co-precipitation technique [2,3]. This technique is still used today as a basis for producting high quality colloid suspensions of magnetic particles in a variety of liquid carrier fluids.

The most commonly used ferrofluid contains spherical magnetic particles with typical size of 10 nm, dispersed in an apolar solvent. Sedimentation of these particles is sufficiently counteracted by Brownian motion to keep them dispersed for years. A prerequisite for such long-term stability is that particles do not aggregate, since aggregates sediment faster and have slower Brownian motion to compensate sedimentation. To prevent aggregation, the colloids can be covered with a thin layer of surfactant, commonly a monolayer of oleic acid (steric repulsion), or the particles are prevented from sticking to each other by electrostatic bilayer (electrostatic repulsion), which makes the particles stable in many liquid carriers (Fig.1). The typical thickness of the surfactant layer is about 2-3 nm.

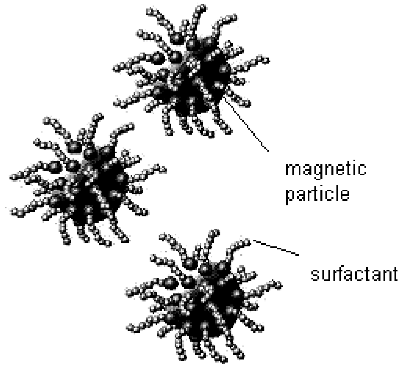

Fig. 1 Schematic sketch of the magnetic particles including their surfactant.

The behavior of such ferrofluids is mainly determined by their magnetic properties. Because of their small size, these magnetic colloids contain a single magnetic domain, and therefore have a permanent magnetic moment proportional to their volume. Although magnetic colloids are ferromagnetic on the molecular scale, they resemble a paramagnet on the colloidal scale, with the major difference that magnetic moments of magnetic colloids are much larger than the moments in a paramagnet (typical values are 10^3-$10^4\mu_B$ for magnetic colloids and order of $1\mu_B$ for paramagnets). Typical number of molecules in magnetic particle of 10nm in diameter is 10^5-10^6 (Fig.2).

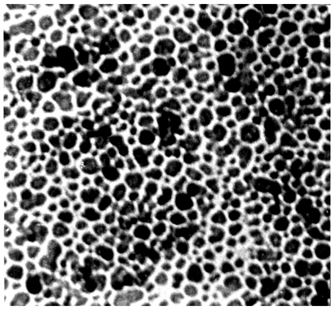

Fig. 2. Typical TEM picture of magnetite particles (1mm=6nm) by TESLA BS 500 microscope normally operated at 90kV and 80 000x magnification by replication technique.

Such a system of particles does not retain any remanent magnetisation as it is superparamagnetic, i.e. the particles have no hysteresis. Due to their superparamagnetic nature, ferrofluids behave as non-magnetic fluids under conditions of zero magnetic field. Each coated particle will behave as a single domain particle and so any rotation of magnetisation is brought about both Brownian and Néel mechanisms. Thermal fluctuation is sufficient to keep the magnetisation vector of particles randomly oriented, such that the net magnetisation of the system is zero. In the presence of a magnetic field, the magnetic moment of the particles will try to align with the magnetic field direction leading to a macroscopic megnetization of the liquid. The magnetisation *M* of the liquid can be described by known paramagnetic behaviour as shown in Fig.3.

An important property of concentrated ferrofluids is that they are strongly attracted by permanent magnets, while their liquid character is preserved. The attraction can be strong enough to overcome the force of gravity. Many applications of ferrofluids are based on this property. Particle size is an important parameter in ferrofluid production. The mean magnetic particle size determine the initial susceptibility of the final fluid and determine the final fluid viscosity.

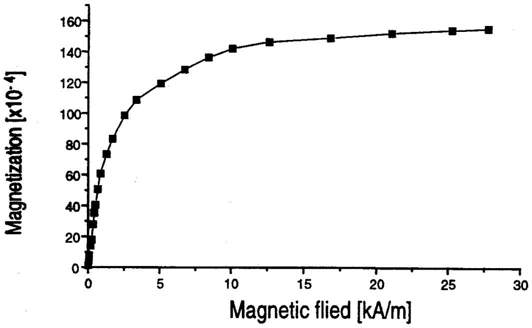

Fig. 3. Typical magnetization curve of a ferrofluid.

In general, the larger is the particle size the greater will be the initial magnetic susceptibility of the fluid and lower the final fluid viscosity. Particle growth can be controlled in a number of ways; for example, variation of precipitation condition or choice of surfactant. The macroscopic properties of ferrofluids not only depend on properties of single particles, but also on their mutual interactions. Some applications depend on the stability of ferrofluids, other depend on their instability. Because of the colloid's Brownian motion, the behavior of magnetic fluid is dictated by thermodynamics, so given the characteristics of the magnetic colloids, the temperature and concentration, the system adapts that minimizes its free energy. The anisotropic nature of magnetic interaction leads to a rich phase behavior of magnetic fluids. For example, because magnetic interaction favors head-to-tail configurations of magnetic colloids, worm-like structure can be expected in dilute solutions of strongly interacting magnetic colloids. In concentrated magnetic fluids, calculations sugest that strong interaction may lead to macroscopic parallel alignment of dipole moments, yielding a liquid permanent magnet. When magnetic fluids are subjected to a magnetic field, the structural changes can occur on microscopic level. Because an external magnetic field aligns the dipole moments of magnetic colloids, it can increase the average interaction strength between magnetic colloids sufficiently to induce aggregation of colloids into concentrated, micron-sized droplets [6]. As the size of such droplets is comparable to the wavelenght of light, the optical properties of ferrofluids depend on the direction and strength of the external

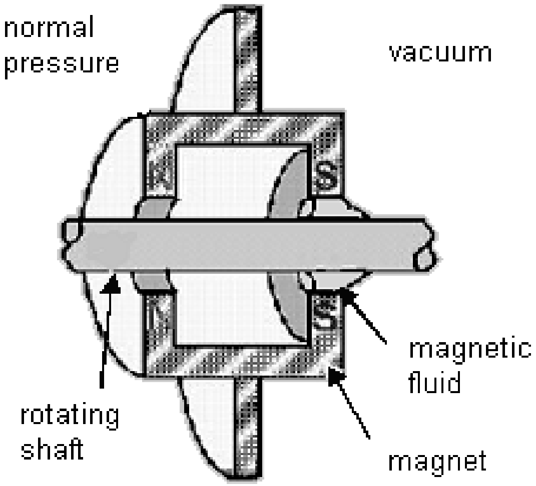

Fig.4 Magnetic fluid sealing of a rotating shaft.

magnetic field. Optical devices employing the strong magneto-optical effect of magnetic fluids are still in development. Industrial applications of magnetic fluids cover a broad spectrum such as magnetic seals in motors, magnetic recording media and biomedical applications such as magnetic resonance contrast media and therapeutic agents in cancer treatment.

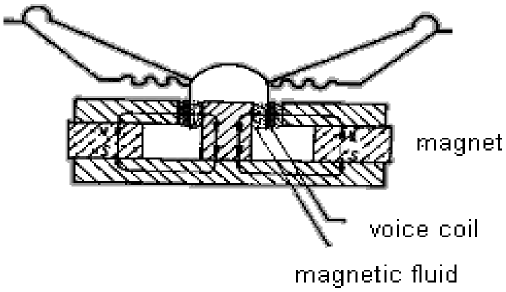

Fig. 5 Magnetic fluid in loudspeaker.

Each potential application requires the magnetic fluids to have different properties.
The magnetic fluid research started in Košice 20 years ago. The main topics under the study are: magneto-optical and magneto-dielectric properties, thermodiffusion, dielectric breakdown, biomedical and biotechnological applications and ferronematics. The aim of this paper is to summarize the main results obtained in these areas. The first part of this review is concerned with different technical applications. The second part deals with possible use of magnetic nanoparticles for biomedical application. Finally, we present the main results of the research in our Institute.

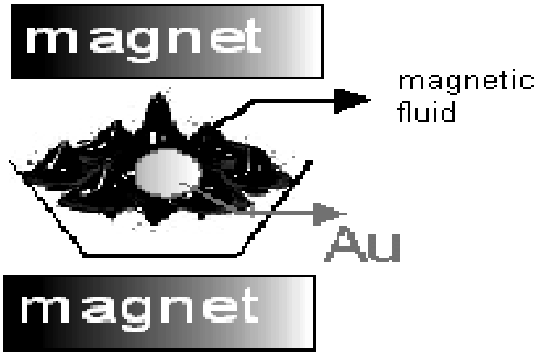

Fig. 6 Separation with magnetic fluids.

2. TECHNICAL APPLICATIONS

The magnetic control of ferrofluids forced strong efforts in the desing of applications using the influence of a magnetic field, in particular the possibility to position the fluid inside a technical device, leading to lot of application for ferrofluids [4]. Some of these reached commercial importance and are widely used in everyday life. Ferrofluids are widely used as lubricating, airtight seals in rotary shafts. A magnetic field gradient keeps the ferrofluids in place, even in case of pressure differences between the two separated compartments. Today, many computer hard disk drives contain a ferrofluid-sealed shaft. Ferrofluids are also used to improve heat dissipation in loudspeaker coils, enabling higher output power (Fig.5).
When non-magnetic objects are immersed in a ferrofluid and subjected to the field gradient of permanent magnet, the object will be effectively repelled by magnet (actually, the ferrofluid is attracted and drives away the object). When combined with a gravitational or centrifugational force opposing the effective magnetic force, this effective repulsion has the same effect as a density gradient of solvent. This principle is used to separate materials into density fractions, for instance in the mining industry or waste processing [5]. Because the effective density of ferrofluids can be much higher than that of ordinary liquids, density-based separation

with ferrofluids can be much higher than that of ordinary liquids. Density-based separation with ferrofluids is also suitable for high density materials such as nonmagnetic metals, diamonds, etc.

3. BIOMEDICAL APPLICATIONS

Magnetic nanoparticles offer some attractive possibilities in biomedicine as they have controllable sizes ranging from a few nanometers up to tens of nanometers, which places them at dimensions that are smaller than or comparable to those of the cell (10-100 μm), a virus (20-450 nm), a protein (5-50 nm) or a gene (2 nm wide and 10-100 nm long). This means that they can get close to a biological entity of interest. Indeed, they can be coated with biological molecules to make them interact with or bind to a biological entity, thereby providing a controllable means of tagging or addressing it. The nanoparticles are magnetic, which means thay obey Coulomb's law and can be manipulated by an external magnetic field gradient. This 'action at a distance', combined with the intrinsic penetrability of magnetic fields into human tissue, opens up many applications involving the transport and immobilization of magnetic nanoparticles, or of magnetically tagged biological entities. In this way they can be made to deliver a package, such as an anticancer drug, an anti-inflamatory drug or clot lysis to a targeted region of the body.

The major disadvantage of most chemoterapies is that they are relatively non-specific. The therapeutic drugs are administered intravenously leading to general systemic distribution, resulting in deleterious side-effects as the drug attacts normal, healthy cells in addition to the target tummour cells. However, if such treatments could be localized, e.g. to the site of a joint, then the continued use of these very potent and effective agents could be made possible. Recognition of this led researchers to propose the use of magnetic carriers to target specific sites (generally cancerous tummours) within the body (Fig.6). In magnetically targeted therapy, a cytotoxic drug is attached to a biocompatible magnetic nanoparticle carrier.

Generally, the magnetic component of the particle is coated by a biocompatible polymer such as PEG, PLA or dextran, although recently inoganic coatings such as silica have been developed. The coating acts to shield the magnetic particle from the surrouding environment and can also be functionalized by attaching carboxyl groups, biotin, avidin, carbodi-imide and other molekules. These molecules then act as attachment points for the coupling of cytotoxic drugs or target antibodies to the carrier complex. The carriers typically have one of two structural configurations: a magnetic particle core coated with biocompatible polymer or porous biocompatible polymer in which magnetic nanopartiles are precipitated inside the pores. The physical and chemical properties of magnetic fluids are strongly influenced by details of the size distribution of dispersed colloidal magnetic particles.

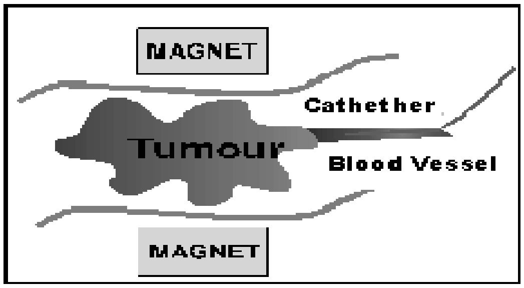

Fig. 7 Magnetic fluid in a cancer therapy

The process of drug localization using magnetic delivery system is based on the competetion between forces exerted on a particle by blood compartement, and magnetic forces generated from magnet, i.e. applied field. When the magnetic forces exceed the linear blood flow rates in arteries (10cms^{-1}) or capillaries (0.05cms^{-1}), the magnetic particles are retained at the target site and may be internalized by the endothelial cells of the target tissue. For this application the use of nanoparticles favour the transport through the capillary systems of organs and tissue avoiding vessel embolism.

The magnetic nanoparticles can be made to resonantly respond to a time-varying magnetic field, with advantageous results related to the transfer of energy from exciting field to nanoparticles. For example, the particles can be made to heat up, which leads to their use as hypertermia agents. Hypertermia is a therapeutic procedure used to rice the temperature of a region of the body affected by malignancy or other growths. The rationale is based on a direct cell-killing effect at temperature above 41-42°C. Modern clinical hypertermia trials focus mainly on the optimization of thermal homogenity at moderate temperatures (42-43°C) in the target volume. The temperature increase required for hyperthermia can be achieved also by using fine iron oxide magnetic particles. The physical principle for which a magnetic material can be heated by action of an external alternating magnetic field are the loss processes that occur during the reorientation of magnetization of magnetic materials with low electrical conductivity. The advantage of magnetic hyperthermia is that allows the heating to be restricted to the tumour area.

Magnetic separation has been succesfully applied to many aspects of biomedical and biological research. In this procedure the magnetic adsorbent is added to a solution or suspension containing the target. This is adsorbed onto the magnetic adsorbent and then the adsorbent with adsorbed target is recovered from the suspension using an appropriate magnetic separator [7].

Magnetic separation has proven to be a highly sensitive technique for the selection of rare tumour cells from blood, and is especially well suited to the separation of low numbers of target cells. This has, for example, led to the enhanced detection of malarial parasites in blood samples either by utilizing the magnetic properties of the parasite or through labelling the red blood cells with an immunospecific magnetic fluid.

These, and many other potential applications, are made avaliable in biomedicine as a result of the special physical properties of magnetic nanoparticles.

4. MAGNETIC FLUID RESEARCH IN KOŠICE

The magnetic fluid research started in Košice 20 years ago. The main topics under the sttudy are: magneto-optical and magnetodielectric properties, thermodifusion, dielectric breakdown, biomedical and biotechnological applications and ferronematics.

4.1. The magneto-dielectric and magneto-optical properties of magnetic fluids

The dielectric constant ε of magnetic fluids is a function of an applied magnetic field and the relative directions of the electric and magnetic field intensities. This is well known as magneto-dielectric and magneto-dielectric anisotropy effect. In our work [8] the magneto-dielectric effect in concetrated suspension of magnetite in mineral oil as a function of the applied magnetic field and of the angle θ between the direction of the electric and magnetic field intensities was studied.

In paper [9] we have studied the absorption of the light in the infrared region in the transverse geometry (magnetic field applied in the plane of magnetic fluid was perpendicular to the light path) as a function of the applied magnetic field H and of the angle θ between the electric vector E of polarized incident light and the magnetic field vector H. The mineral oil based magnetic luid with Fe_3O_4 magnetic particles with surfactant oleic acid has been used. The experimental results showed, thet the change of the absorption coefficient ΔA with H and wavenumber k is zerofor the angle $\theta = 54.73°$. This was confirmed by measurements in five mineral oil based magnetic fluids.

4. 2 The termodifusion in magnetic fluids

The light induced heating of fluids can give rise to interesting phenomena, which depend upon the illumination character and the type of illuminated magnetic fluid. When two intense coherent beams pass through an absorbting fluid, the periodic heating and following periodic modulation of the refractive index occurs in their interference field. This manifests in diffraction of the light beams passing through the fluid (selfdifraction on phase grating). When a colloidal fluid is illuminated this way, not only refractive index space modulation, but also the redistribution of colloidal particles appears, connected with space modulation of absorption coefficient and thus with the diffraction of the light on created amplitude grating. The redistribution of colloidal particles is the result of the thermodifusion, a flow of colloidal particles invoked by temperature gradient. This process is characterized by Soret constant $S=D_T/D_{dif}$, where D_T is the thermal diffusion coefficient and D_{dif} is the particle translation diffusion coefficient. The sign of the Soret constant represents the direction of the particle diffusion – positive if colloidal particles migrate against the temperature gradient direction, negative if the direction of the migration is identical with the temperature gradient direction. Very interesting phenomena can be observed if a thin sample of colloidal magnetic fluid with negative Soret constant is exposed to intensive illumination. The large changes of particle concentration arise which influence the light absorption coefficient and this way also the heating distribution. In such a case a fluctuation of absorption may spontaneously increase what can give rise to a self-structuralisation of the particle density. The self-difraction as well as self-structuralisation were studied in magnetic fluids containing Fe_3O_4 particles dispersed in various media. In effort to explain the observed phenomena we have tried to give a more detailed description of the thermodiffusion process in colloidal fluids [10, 11]. This theoretical description led also to the development of a method for the determination of the distribution of fine magnetic particles hydrodynamic diameters in magnetic fluids.

4. 3 The dielectric breakdown strength of magnetic fluids

Highly refined mineral oils, so called transformer oils, are typically used for insulation and cooling in electromagnetic devices, such as power transformers. It has been shown that magnetic fluids based on these oils can substantially improve the heat transfer and AC – dielectric breakdown strenght in these devices. The DC impulse breakdown voltage in such magnetic fluid was found to be nearly independent on the polarity of the needle impulse, as for pure oil there is a big difference between the breakdown voltages corresponding to opposite polarities. Since only the lower value of the impulse voltage can be relied upon for the safe operation of the transformer, magnetic fluid increase the limit from 78kV to 108kV.

4. 4 The drug immobilization to fine magnetic particles

One of the prerequisites for success of the application of drug targeting for treatment of localised diseases is development of an effective method to transport the drug to the target site in organism. In recent years, there has been growing interest in magnetic substances for the creation of magnetic pharmaceutical preparations. In particular, magnetite can be used as a drug carier, which makes it possible to create magnetically guided drugs. Such drugs can be delivered to a target organ under the action of an external magnetic field. The superparamagnetic property of fine magnetic particles is very important from a practical point of view because it means that magnetic particles can be precisely transported, positioned and controlled in desirable parts of blood vessels or hollow organs by external magnetic field. In our work [12] an attempt was made to link protein molecules bovine serum albumin (BSA), glucose oxidase (GOD), chymotrypsin, streptokinase and dispase) directly to magnetic particles using 1-[3-Dimethylamino)propyl]-3-ethylcarbodimide hydrochlorid (CDI) as the coupling agent. Different values of pH and ratios of magnetic particles to protein were studied in order to establish the optimum conditions for immobilization. The direct coupling of enzymes or bioactive molecules to the magnetic particles has a number of potential advantages. The lack of polymer coat results in smaller particles, thus increasing the ratio of surface area to volume, allowing a greater response to any magnetic field. Magnetic particles (Fe_3O_4) were prepared by co-precipitating ferric and ferrous salts in alcaline solution. The amount of magnetic particles in a given volume of the ferrofluid was estimated by thermogravimetry and by magnetic measurements of magnetic curves (VSM magnetometer). The particle size distribution was determined by electron microscopy and magnetic measurements. The particles were found to have a lognormal particle size distribution with mean diameter of 10 nm. The general procedure of immobilization of proteins and enzymes to fine magnetic particles can be described as follows: to study pH effect of the reaction mixture on the coupling reactions, the solution of proteins or enzymes and CDI were prepared in the buffers (sodium or potassium phosphate) of various pH from 4.5 to 6.5. The reaction mixtures which contained magnetic particles (A), proteins or enzymes (B) and CDI (C) with various mass ratios A:B:C were prepared. Then the reaction mixtures were shaken for a period of 24 h at room temperature. After the incubation period, samples were placed on the top of a bar magnet where sedimentation of magnetic particles occurred within 2 min. The protein estimation was carried out using Bradford's dye binding assay. Protein content of each of the samples and controls (containing no magnetic particles and CDI) was estimeted before incubation. Following incubation, protein content of the supernatants, washings and the controls was determined. The difference in the protein content of reaction system before and after incubation corresponded to the amount of protein coated onto magnetic particles. The immobilization was confirmed by the FTIR spectroscopy and electron microscopy. The optimal conditions for the immobilization of the various proteins and enzymes to fine magnetic particles depend on the kind of immobilized proteins and enzymes, the pH of the reaction mixture and the ratio of each reagents in the reaction mixture, respectively. The usefulness of the presented method is in medicine and biotechnology. One of the important applications is the treatment of coronary thrombosis and peripheral arterial occlusions. By drawing magnetic streptokinase to the target site of a thrombosis by applying powerful magnetic fields to the patient it should be possible to lyse the clots using reduced quantities of drug and to avoid the unwanted side effects of high doses of streptokinase.

There are several direction in the creation of magnetically guided drugs. The one of them is based on the use of stabilizing agent such as dextran, PVA, pluronic etc., whereby a polymeric coat with a drug is formed on the surface of a magnetic particles. In our work we used as a stabilizing agent pluronic, then a polymer shell was formed from biodegradable polymer PLA (Poly D,L – lactic acid). The polymer magnetic nanospheres were prepared according to a modified nanoprecipitation method [13]. PLA polymer and specified quantity of drug indometacin (anti-inflammatory drug) were accurately weighed and dissolved in the mixed organic solvent of acetone (miscible with water) and chloroform (immiscible with water). The contents were allowed to stand at room temperature for 15 minutes with occasional vortexing to allow complete solubilization of the drug and the polymer. Then the organic phase was added dropwise into the aqueous phase containing of 0.5ml the magnetic fluid with the concentration Fe_3O_4 = 1mg/ml, 10ml Pluronic (1.25mg/ml) and 10ml of phosphate buffer of pH =7.4 and stirred magnetically at room temperature until complete evaporation of the organic solvent had taken place, allowing the formation of a turbid nanoparticles suspension. With the aim of analyzing of the prepared nanospheres, infrared spectra of materials were obtained, as shown in Fig.7. The measurements were performed by the KBr pellet method in the range from 4000 to 400 cm^{-1}. In this method, the solid sample is finaly pulverized with pure, dry KBr, the mixture is pressed in a hydraulic press to form a transparent pellet, and the spectrum of the pellet is measured.

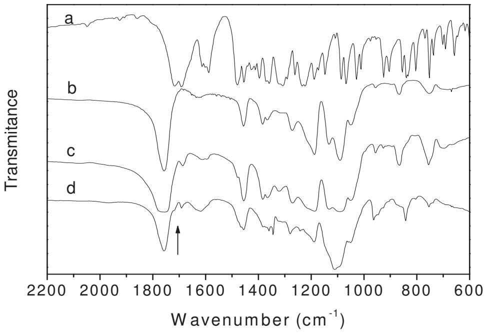

Fig.8 Infrared spectrum of pure indometacin (a), PLA (b), encapsulated indometacin in PLA (c) and magnetic nanoparticles with indometacin encapsulated by PLA (d)

Figure 7 shows typical spectra of pure indometacin (a), PLA (b), prepared polymeric nanospheres with indometacin (c) and magnetic particles with indometacin incorporated in PLA in the range from 2200 to 600 cm^{-1}. The spectrum of indometacin display a characteristic absorption doublet at 1695 and 1715 cm^{-1} (C=O streching) and PLA display a characteristic absorption band at 1758 cm^{-1} (C=O stretching).

As observed, the characteristic absorption band of PLA and indometacin observed at 1758 cm^{-1} and 1695 cm^{-1}, respectively appears in the spectrum of the composite of PLA spheres with indometacin. Note, however, that the single PLA band at 1758cm^{-1} is broaden suggesting a slight contribution of the 1715 cm^{-1} band of indometacin. As shows fig.7, the spectrum of magnetic nanoparticles with indomethacin loaded nanospheres (d) is very similar to that of PLA indometacin loaded spheres (c). The presence of a characteristic bands of PLA and indometacin in infrared spectra of obtained complex magnetite – PLA - indometacin confirmed successful encapsulation the indometacin and magnetic particles into PLA.Nanotechnology is beinning to allow scientists to work at the cellular and molecular levels to produce major advances in the life sciences and healthcare. Real applications of nanostructured materials materials in life sciences are uncommon at the present time, but the excellent properties of these materials provide a very promising future for their use in this field.

It is now generally recognized that nanotechnologies and biosciences will be one of the leading and most promising areas of research and development in the 21st century. We hope this review made clear that magnetic fluids could play a very important role in these developments.

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