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Tailored and Functionalized Magnetite Particles for Biomedical and Industrial Applications

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1. Introduction

Magnetic particles have a significant role in nanotechnology due to their surface properties and their applicability in physical and chemical processes like ionic exchange, specific complexation, biocompatibility and bioactivity, capacity of selection and transport for cells and chemical compounds (Safarik & Safarikova, 2002).

Magnetite is an interesting superparamagnetic nanomaterial, considered as a challenge by the modern research related magnetic applications, due to its high susceptibility at oxidation as compared to other magnetic compounds (Cornell & Schwertmann, 1996).

The name "superparamagnetic" refers to those particles that in presence of magnetic field are attracted and in absence of the magnetic field the particles don't have residual magnetism. The importance of magnetite particles is related to their most important properties - magnetic and catalytic. These properties are strongly dependent on the selected method of preparation.

These magnetic particles are used in many applications that involve their immobilization and transport in the presence of magnetic field, or magnetically tagged biological entities due to the intrinsic penetrability of magnetic fields into human tissue.

The chapter describes the most important approaches in the preparation of magnetite particles, as presented in the literature. An introspective view of biomedical, industrial and catalytic applications of magnetite micro- and nanoparticles is also reported.

The magnetite particles are usually attracted to each other. Electrostatic or steric stabilization represent the main step in obtaining "core-shell" magnetic particles with magnetite core and a shell formed by different surfactants. We summarize here some examples and also some data previously reported by our group (figure 1) on the preparation of magnetite particles coated by different Si-containing compounds (monomers and polymers) (Durdureanu-Angheluta et al., 2008; Durdureanu-Angheluta et al., 2009; Durdureanu-Angheluta et al., 2010; Pricop et al., 2010).

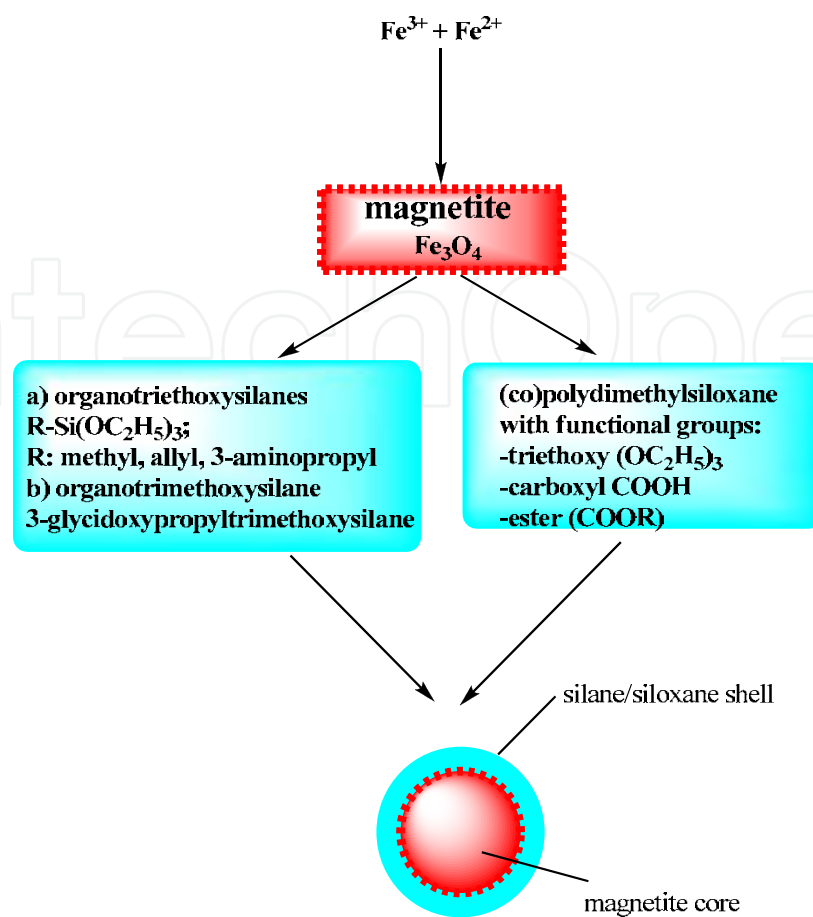


Fig. 1. Schematic representation of the preparation of core-shell magnetite particles with different Si-containing compounds.

2. Synthesis methods

Magnetite particles are quite intriguing, due to their catalytic and magnetic properties strongly dependent on the chosen synthesis method. Several methods are described in the literature, but in the present manuscript we are going to focus on the most common ones: iron salts co-precipitation (Massart, 1981; Martinez-Mera et al., 2007), sol-gel reaction, micro-emulsion (Woo et al., 2003), reaction in mass without solvent (Ye et al., 2006), polyols process (Feldmann & Jungk, 2001), decomposition of iron pentacarbonyl (Shafi et al., 2001), etc.

2.1 Co-precipitation

The synthesis by co-precipitation is the easiest way to obtain iron oxides like magnetite (Fe_3O_4) (Laurent et al., 2008) or like maghemite ($\gamma\text{-Fe}_2\text{O}_3$) and has shown to be a highly economic and versatile method. We used the method described by Massart (Massart, 1981) involving the co-precipitation of two iron salts $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$, both prepared in HCl, with the consequent addition of a NH_4OH solution, vigorous stirring and under anoxic conditions, at room temperature. The shift of the initially orange color of the solution to black indicates the formation of magnetite particles. The surface of magnetite particles, in

contact with a neutral aqueous solution is able to adsorb OH^- and H^+ , respectively, giving an OH^- -rich surface (Durdureanu-Angheluta et al., 2008).

The reaction for obtaining magnetite is given below:



The disadvantage of the co-precipitation process is that the control of particle size distribution is limited, due to kinetic factors that are controlling the growth of the crystal. Two stages are involved in the co-precipitation process (Cornell & Schwertmann, 1996; Boistelle et al., 1988; Sugimoto, 2003; Cornell et al., 1991; Gribanow et al., 1990): nucleation that occurs when the concentration of the species reaches critical supersaturation, and a slow growth of the nuclei by diffusion of the solutes to the surface of the crystal. To produce monodisperse iron oxide nanoparticles, these two stages should be separated. Nucleation should be avoided during the period of growth (Tartaj et al., 2006).

An alternative route to obtain magnetite particles by co-precipitation is the method described by Kim et al. (Kim et al., 2001) and Koneracka et al. (Koneracka et al., 2002) involving the preparation of the two solutions of ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) and ferrous sulphate (FeSO_4) by dissolution in a HCl solution at room temperature under vigorous stirring using a mechanical stirrer. The solutions are mixed and an aqueous dispersion of particles is obtained after adding a solution of NaOH under sonication or under vigorous mechanical stirring.

Magnetic particles are prepared, also, using $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ salts in aqueous solutions, both added in a polymeric starch matrix at 80°C under nitrogen atmosphere and vigorous stirring (Jiang et al., 2009). NaOH should be added to the mixture until reaching a basic pH (9-11) and the black precipitate is neutralized with HCl .

2.2 Sol-gel reaction

The reaction takes place at the water/oil interface. An aqueous solution containing $\text{Fe}^{2+}/\text{Fe}^{3+}$ salts is added to an organic solvent containing the stabilizer. The sol-gel reaction is performed at room temperature and is based on the hydroxylation and condensation of molecular precursors in solution and leads to a three-dimensional metal oxide network, the wet gel. Heat treatments are further needed to acquire the final crystalline state (Liu et al., 1997; Kojima et al., 1997). The properties of the gel are dependent on the structure created during the sol stage of the sol-gel process.

Sol-gel nanocomposite materials ($\text{Fe}_x\text{O}_y\text{-SiO}_2$) were obtained using alkoxide and aqueous routes (Raileanu et al., 2003). The structure and properties of the prepared particles were compared for different precursors of silica (tetramethoxysilane, methyltriethoxysilane, colloidal silica solution, etc.).

Magnetite-based ferrofluids were synthesized through a modified sol-gel method (Gamarra et al., 2005). They precipitated iron oxyhydroxy in water in the presence of a surfactant (Renex), and next they partially reduced Fe^{3+} to Fe^{2+} by a mild drying process in N_2 atmosphere, leading to the formation of magnetite.

2.3 Free-solvent synthesis

The free-solvent synthesis of magnetite is an economical and non-toxic method. The solid iron salts ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$) and a solid base (NaOH) are used directly with the surfactant and mixed together using a mortar and a pestle (Ye et al., 2006). The synthesis procedure takes place at room temperature in a glove box filled with argon gas. This method is easy, without any advanced techniques and toxic solvents. The reaction duration is about a few minutes and the high yield magnetite obtained is monodisperse.

2.4 Other methods

Polyol process

Polyols are known to reduce metal salts to metal particles and to be good solvents for various inorganic compounds. This is why the synthesis of nano- and microparticles with well-defined shapes and controlled sizes can be performed by the polyol process, also (Fievet et al., 1989). The metal precursor is heated to a given temperature which cannot be higher than the boiling point of the polyols, with generation of intermediates that are reduced to metal nuclei and nucleate to form metal particles. Polyols act like solvents due to their high dielectric constants and like surfactants, preventing the particles aggregation. Their high boiling points offer the advantages of operating on a large temperature interval (from 25°C up to the boiling point). The “core-shell” PVP (poly(vinylpyrrolidone))-nanoparticles can be obtained in one-pot polyol process by reduction of $\text{Fe}(\text{acac})_3$ with 1,2-hexadecanediol in the presence of the PVP polymer surfactant in octyl ether (Liu et al., 2007). These particles are promising for biomedical applications like MRI agents and biosensors. The magnetite nanoparticles prepared by this method have a long-term stability and are easily coated by various materials to generate multifunctional nanoparticles. Non-aggregated magnetite nanoparticles have been already synthesized in liquid polyols at elevated temperature (Cai et al., 2007). Highly performance magnetite nanoparticles have been also obtained by a modified polyol process. For that, only one iron rich precursor has been used and no further reducing agent and surfactants were required. The magnetite nanoparticles can be coated in situ by hydrophilic polyol ligands, and the hydrophilic triethylene glycol magnetic nanoparticles are easily dispersed in water and other polar solvents. This method is a good alternative to produce superparamagnetic water-soluble magnetic nanoparticles and can replace the co-precipitation method.

Micro-emulsion

Emulsions are thermodynamically stable and consist of two different immiscible liquids (like oil - organic solvent and water) (Bagwe et al., 2001). The emulsion is stabilized by adding a surfactant. The size of magnetite particles synthesized by micro-emulsion method is several nanometers in diameter (López Pérez et al., 1997), except in some specific cases (20-30 nm) (Hirai et al., 1997). This method is difficult to control, the obtained yield is low and the particles are polydisperse. The commonly used method is the one that involves water-in-oil micro-emulsion and also includes a reverse micelle system (Ganguli et al., 2003). For the synthesis of Fe_3O_4 , the precipitation technique consisting of alkalization of a solution of metal salt and hydrolysis in micro-emulsions (Boal et al., 2004; Hirai et al., 1997) is commonly used, as well as biosynthetic routes (Matsunaga, 1998).

Magnetite/silica particles were prepared by reverse-micelle micro-emulsions in an organic phase such as cyclohexane and heptane (Yang et al., 2004; Santra et al., 2001).

Fe₃O₄ nanoparticles in poly(organosilsesquioxane) with sizes of 4-15 nm were prepared by the one-pot synthesis using reverse micelle method (Ervithayasuporn et al., 2009). The ferrofluid droplets were in situ encapsulated, via polycondensation of molecularly self-assembled octenyltrimethoxysilane.

Sonochemical decomposition of iron pentacarbonyl

Magnetite nanoparticles were obtained by a sonochemical micro-emulsion polymerization process of n-butyl methacrylate (BMA) monomer (Teo et al., 2009). Iron tri(acetylacetonate), 1,2-tetradecanediol, oleic acid, and oleyl amine were mixed in benzyl ether, under vigorous stirring. The mixture was heated gradually under nitrogen atmosphere and the black-colored mixture was then cooled to room temperature by removing the heat source. The pre-formed particles were then encapsulated within the host poly(BMA) latex particles with 120 nm diameter and low size dispersion. The distribution of magnetite particles over the polymer particle population and within each polymer particle was nevertheless rather heterogeneous and the ratio of magnetite particles per polymer particle was determined as being equal to 50.

3. Influence of Fe²⁺ onto magnetite properties

The stoichiometric factor of Fe²⁺ and Fe³⁺ is important for the magnetic properties of the obtained particles (coercivity, crystallinity, sorption capacity, etc.) (Gorski et al., 2010). The Fe²⁺ chemical species direct the magnetite particles synthesis kinetics and their composition (Tronc et al., 1992). The Fe²⁺/Fe³⁺ molar ratio is highly important if one focusses on obtaining magnetite particles with specific properties. Thus, a Fe²⁺/Fe³⁺<0.1 molar ratio is too small to achieve a stable solution. In this context, if the content of chemical species of Fe²⁺ is low, goethite (α-FeO(OH)) is obtained as the only stable product. The use of the Fe²⁺/Fe³⁺>0.1 molar ratio is favorable for obtaining magnetite instead of goethite.

The influence of the Fe²⁺/Fe³⁺ molar ratio on the characteristics of magnetite particles obtained by co-precipitation of Fe²⁺ and Fe³⁺ (composition, size, morphology and magnetic properties) was studied by Jolivet (Jolivet et al., 1992). Chemical species in different proportions were precipitated with ammonia solution to pH~11. The analysis of the products obtained with different Fe²⁺/Fe³⁺ molar ratios, in the range 0.10-0.50, concluded that for values lower than 0.30 two different phases coexist:

(a) the first phase contains particles of 4 nm in diameter, with oxyhydroxy (FeO(OH)) surface functional groups and low Fe²⁺ content, reflected by the ratio Fe²⁺/Fe³⁺~0.07;

(b) the second phase is characterized by increased content of Fe²⁺ (Fe²⁺/Fe³⁺~0.33 molar ratio), the final product is magnetite with increased particle sizes. It was noticed that the share of this phase increases with increasing the Fe²⁺/Fe³⁺ molar ratio. Thus, for values greater than 0.35, the product is found only in the second phase.

In conclusion, for a 0.5 Fe²⁺/Fe³⁺ molar ratio homogenous magnetite particles of uniform size and composition are more likely to be obtained. The order of addition of ionic species (Fe²⁺ and Fe³⁺) in co-precipitation reaction does not influence the final characteristics (size, composition) of the obtained particles.

The influence of ionic species concentrations on the properties of magnetite particles was also followed. It was noted that the $\text{Fe}^{2+}/\text{Fe}^{3+}$ molar ratio was a determining factor in obtaining sub-micron sizes, while by increasing the ratio, the mean diameter of the magnetic particles increased, but unfortunately the yield decreased (Babes et al., 1999).

4. Sterically functionalized magnetite core-shell particles

Magnetite surface chemistry

Magnetite particles act as Lewis acids in aqueous systems and coordinate water or hydroxyl groups. This is why the particles surface chemistry is highly dependent of pH value; at low pH the surface of the particles is protonated and at high pH the surface is negatively charged (Figure 2). The hydroxyl groups from magnetite surface are the reactive parts, which can react with acid or base. The HO^- groups are reacting with other organic or inorganic anions and may adsorb protons or cations, also.

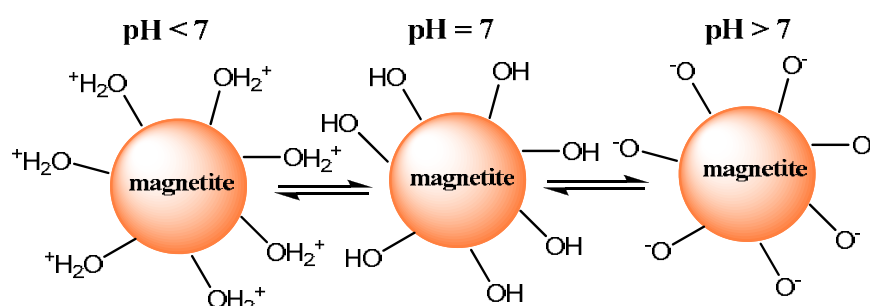


Fig. 2. Magnetite particles behavior at low/high pH.

Magnetic ferrofluids based on magnetite particles have a high capacity of agglomeration due to the fact that two types of forces co-exist: attraction and repulsive forces. The stabilization of the magnetic particles requires the control of these forces. To obtain stable magnetic dispersions, the attraction forces between magnetic particles must be overcome by electrostatic or steric stabilization (Figure 3).

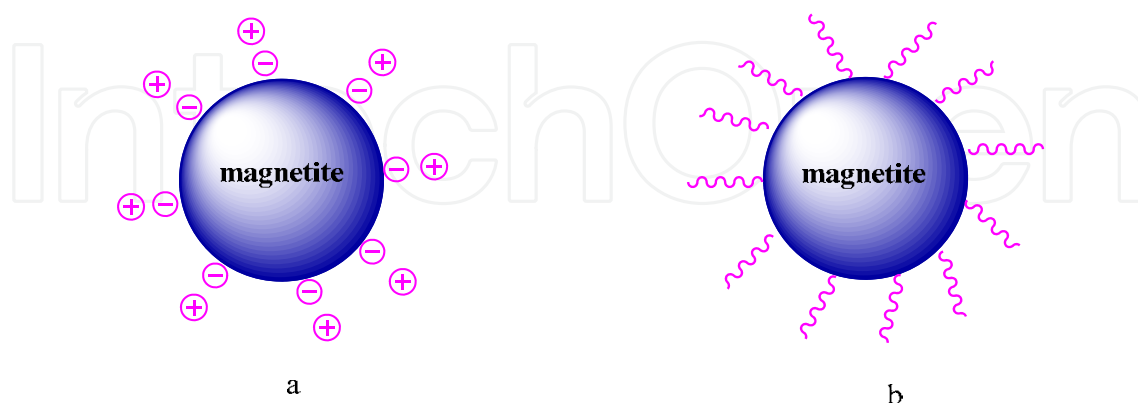


Fig. 3. Electrostatic (a) and steric (b) stabilization of the particles.

In order to stabilize magnetite particles, monomers/polymers with functional groups that can bind to the surface of the particles and act as surfactants can be used. The rest of the chain is solvated in dispersion medium or in a fluid. The process, known to be entropic or

steric, refers to the inhibition of particles aggregation by an entropic force, which appears when the particles are closed to each other. As surfactants monomers, homopolymers, block copolymers and polymers with terminal functional groups can be successfully used. After the coating process, these particles are re-suspended in proper solvents and form homogenous suspensions named ferrofluids (Shen et al., 2004).

Magnetite particles precursors	Coating agent	Diameter (nm)	Type of linkage	Reference
Fe(CO) ₅	Polyethylene, Polystyrene, Polyisobutylene - all three functionalized with tetraethylenepentamine	8-50	Hydrogen bond	Burke et al., 2002
FeCl ₂ •4H ₂ O FeCl ₃ •6H ₂ O	Oleic acid, Stearic acid, Linoleic acid	5.7-9.3		Korolev et al., 2002
FeSO ₄ FeCl ₃	N-oleoylsarcosine	8.1-20.7		Xu et al., 2004
FeCl ₂ •4H ₂ O FeCl ₃ •6H ₂ O	Double shell with oleic acid - Pluronic F-127, and then entrapped the doxorubicin	193 (DLS) 11 (TEM) 9.2 (XRD)		Jain et al., 2005
FeCl ₃ •6H ₂ O FeSO ₄ •6H ₂ O	Xylan	25.26±0.42 μm		Andriola Silva et al., 2007
FeCl ₂ •4H ₂ O FeCl ₃ •6H ₂ O	Carboxyethylsilanetriol sodium salt solution	10 (magnetite) 100 (core-shell particles)		Kobayashi et al., 2008
FeCl ₂ •4H ₂ O FeCl ₃ •6H ₂ O	Ricinoleic acid and Poly(lactic-co-glycolic) acid (PLGA)	140-230		Furlan et al., 2010
FeCl ₂ •4H ₂ O FeCl ₃ •6H ₂ O	Double shell: silica (SiO ₂ /Fe ₃ O ₄) and aluminosilicate	100		Chang et al., 2009
FeCl ₃ Na ₂ SO ₃	Ethoxy-PEGsilane	10 (magnetite)	Covalent bond	Zhang et al., 2002
	N-hydroxysuccinimide PEG-fluorescein Folic acid		Covalent bond Hydrogen bond	
FeCl ₂ •4H ₂ O FeCl ₃ •6H ₂ O	3-aminopropyltrimethoxy silane (APTMS) and monomethoxy-poly(ethylene glycol) (MPEG) (CH ₃ CH ₂ O) ₃ -Si-MPEG	200	Covalent bond	Kim et al., 2002
FeCl ₃ •6H ₂ O FeSO ₄ •7H ₂ O	3-aminopropyltrimethoxy silane (APTES) and Polyethylene glycol diacid (HOOC-PEG-COOH)	40-60		Feng et al., 2008
FeSO ₄ •7H ₂ O	Tetraethoxyorthosilicate (TEOS) (OEt) ₄ Si 3-aminopropyl triethoxysilane (APTES) CS ₂ , NaOH and 2-propanol	50-80		Girginova et al., 2010

Table 1. Core-shell magnetite particles.

The synthesis methods for magnetic particles with magnetite core and monomer/polymer shell, named core-shell magnetic particles, are presented in the literature (Table 1). The surfactant used must have functional groups to interact by hydrogen or covalent bonds (Figure 4) with the hydroxilic groups from pre-formed magnetite surface (trialkoxysilanes or compounds with carboxylic groups) (Kazufumi et al., 2008; Wormuth, 2001; Mondini et al., 2008; Shukla et al., 2007; Wilson et al., 2005) and other functional groups to permit their dispersing in a transport fluid (aminic, epoxy, vinyl groups, etc.).

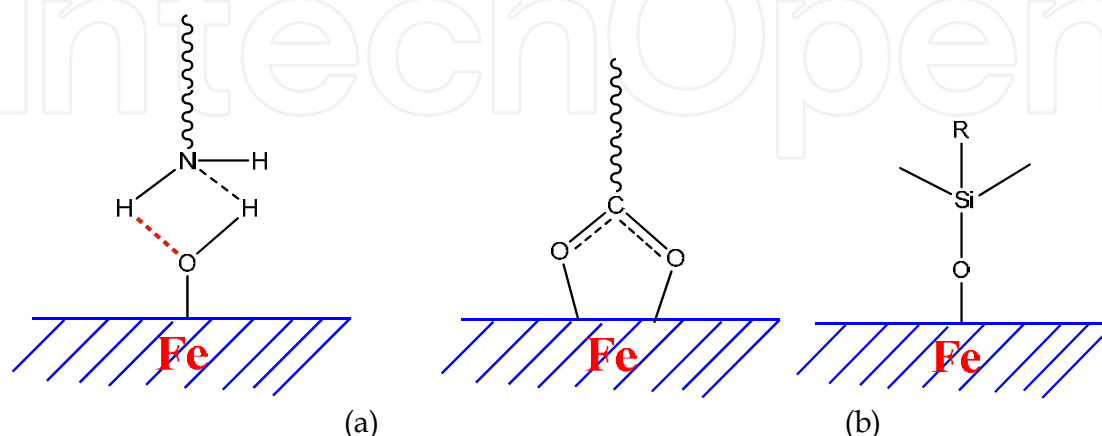


Fig. 4. Schematic representation of hydrogen bond (a) and covalent bond (b) between core and shell.

5. Applications of micro- and nanomagnetite particles

The high interest for magnetic particles (ferrofluids, nanospheres and microspheres) can be explained due to their various applications, e.g., in magnetic storage devices or magnetic cell separations (Goya et al., 2003). The magnetic particles were also tested for cancer treatment and as drug and nucleic acids carriers (Lubbe et al., 1996).

Top domains for multiple applications of micro- and nanoparticles with magnetic properties are biomedical applications, catalysis and industrial applications.

5.1 Biomedical applications

Magnetite nanoparticles represent an intriguing material for various researchers, due to their biomedical applications in the treatment of solid tumours (Gomez-Lopera, 2006) or contrast agents (Frija et al., 2004). In order to be efficient in biological applications, colloidal suspensions of magnetic particles must have long-time stability and the magnetic core must respond to an external magnetic field that directs the particle to a desired location. Colloidal stability is influenced by the size, structure and composition of the particle with a narrow dimensional controlled polydispersity. All these requirements lead to magnetic fluids that flow through the capillaries that provide a maximum diameter of 1.4 μm . The colloidal stability of magnetite particles is insured by coating the magnetic core with a monomer or (co)polymer, as surfactant, to have the particles dispersed in a carrier fluid.

Magnetite (Fe_3O_4) nanoparticles that exhibit excellent magnetic saturation ($\sim 78 \text{ emu/g}$) are desirable for these applications due to the strong ferromagnetic behaviour, lower sensitivity to oxidation and relatively low toxicity as compared to many other materials (e.g., iron,

nickel and cobalt) (O'Brien, 2003). Magnetite has been intensively studied in the past decades due to its application in biotechnology and biomedicine: cancer treatment (Jordan et al., 2001, Jordan et al., 1999, Herrera et al., 2008), sensors, catalysis, storage media, clinical diagnosis and treatment of some diseases (Torchilin et al., 2001, Jordan et al., 2001).

Especially for biomedical application, the particles must present some specific properties like:

1. Magnetic properties

In order to respond to an external magnetic field magnetic particles must have a large saturation magnetization, an important characteristic for targeted drug delivery systems and contrast agents for MRI (magnetic resonance imaging) (Bulte, 2006, Modo et al., 2005, Burtea et al., 2005, Boutry et al., 2006, Babes et al., 1999, Sonvico et al., 2005, Corot et al., 2006, Corot et al., 2007)). In the presence of an external magnetic field the magnetic moments are oriented with the field (Figure 5). Magnetic nanoparticles modified with organic molecules and used for biomedical applications can be magnetically controlled by applying an external magnetic field.

2. Stability in colloidal dispersions

The stability of the particles can be enhanced by coating with biocompatible surfactants (Ma et al., 2003) capable of interactions with hydroxyl groups on the magnetite surface and ensuring the ferrofluid stability. The coating agent should have specific functional groups for further functionalization for specific applications.

3. Nano- sized dimensions

The magnetic particles with biomedical applications must have controllable sizes ranging from a few nanometres up to tens of nanometres, smaller than those of a cell (10–100 μm) (Pankhurst et al., 2003), a virus (20–450 nm), a protein (5–50 nm) or a gene (2 nm wide and 10–100 nm long) because they can 'get close' to a biological entity of interest. Magnetic particles encapsulated into a polymer matrix with dimensions more than 1 μm or sub-micron is more effective in the bloodstream through the veins or arteries (Lazaro et al., 2005).

4. Oxidation stability

Metals such as Fe, Co and Ni, which oxidize easily, are not recommended in biomedical uses due to the higher probability of oxidizing inside human bodies. This problem can be solved using magnetite particles that not only present a greater stability to oxidation, but can be coated with different specific surfactants, increasing their resistance to oxidation.

The polymers used as surfactants can be synthetic or natural molecules able to yield stable colloidal suspensions designed for drug delivery. Nevertheless, synthetic polymers have the advantage of high purity and reproducibility over natural polymers.

The colloidal magnetic particles obtained for biomedical application (Asmatulu et al., 2005) as targeted drug delivery will have to pass through a few mechanisms of releasing drug molecules: diffusion, degradation of polymeric shell, swelling of the shell followed by diffusion of active principle outside the magnetic particle. The diffusion involves drug

molecules dissolving in body fluids around or within the particles and migration of the drug away from the particles. The degradation mechanism occurs when the polymer chains are hydrolysed into lower molecular weight species and release the drug trapped by the polymer chains. The coated particles, when inside the body, swell to increase pressure, and the drug diffuses from the polymer network into the body.

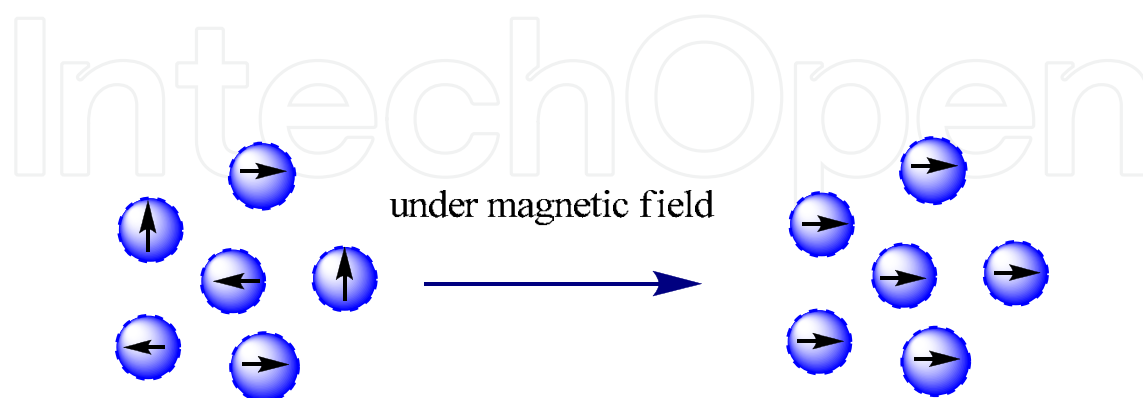


Fig. 5. Magnetite particles in presence/in absence of magnetic field.

Magnetic nanoparticles coated with different organic compounds offer a high potential for numerous biomedical applications, such as cell separation (Kuhara et al., 2004), automated DNA extraction (Yoza et al., 2003), gene targeting (Plank et al., 2003), drug delivery (Lazaro et al., 2005), magnetic resonance imaging (Glech et al., 2005), and hyperthermia (Pardoe et al., 2003). After coating with an antibody, they can be used for immunoassays (Chemla et al., 2000) or small substance recovery (Tanaka et al., 2004). DNA or oligonucleotides immobilized on magnetic particles have been used for DNA hybridization analyses to identify organisms (Matsunaga et al., 2001) and single-nucleotide polymorphism analyses for human blood (Maruyama et al., 2004).

Magnetic particles coated with immunospecific agents have been successfully bound to red blood cells (Molday et al., 1982, Tibbe et al., 1999), lung cancer cells (Kularatne et al., 2002), bacteria (Morisada et al., 2002), urological cancer cells (Zigeuner et al., 2003). Coupling of homing peptide labeled with 5-carboxyl-fluorescein (FAM-A54) and magnetite coated with starch chains functionalized with homing peptide was performed by Schiff's reaction (Jiang et al., 2009). This magnetic fluid has great potential applications in diagnostics and therapeutics of human tumours.

Nano-sized iron oxide particles (Fe_3O_4) were coated with a lipid layer and then functionalized with biotin for further protein ligands. These particles were then used for binding streptavidin–fluorescein isothiocyanate generating a conjugate used to investigate their magnetic and fluorescent activity into cells (Beckera et al., 2007).

The magnetic particle application for hyperthermia goes back to 1957 (Gilchrist et al., 1957). The authors heated up various damaged tissue samples with 20–100 nm size particles of $\gamma\text{-Fe}_2\text{O}_3$ exposed to a 1.2 MHz magnetic field. Since then the scientists have been developing numerous methods and schemes using different types of magnetic materials, different field strengths and frequencies of encapsulation and delivery of the

particles (Mosso et al., 1973, Rand et al., 1976, Gordon et al., 1979, Rand et al., 1981, Borrelli et al., 1984, Hase et al., 1990, Suzuki et al., 1990, Chan et al., 1993, Matsuki et al., 1994, Mitsumori et al., 1994, Suzuki et al., 1995, Moroz et al., 2001, Jones et al., 2002). The hyperthermia procedure involves the dispersion of the magnetic particles throughout the target tissue, followed by applying an AC magnetic field with specific parameters (frequency, strength); the magnetization of the particles is continuously reversed, which translates into a conversion from magnetic to thermal energy and causes the heating of particles. The tumour is destroyed if the temperature is maintained above the therapeutic threshold of 42°C for 30 min or more.

The bisphosphonate modified magnetite nanoparticles were used as agents to remove uranyl ions (e.g., UO_2^{2+}) in vivo from water or blood (Wang et al., 2006). The authors synthesized a bisphosphonate derivative, DA-BP, which modifies the magnetite nanoparticles for the successful decorporation of the radioactive metal from blood due to the chelating effect (Fukuda, 2005). Starting from this study, a protocol that allows the detection and recovery of other heavy metal toxins from the biological systems has been developed and perfected.

Coated magnetite particles with magnetic resonance, fluorescent imaging and drug delivery applications were obtained (Kim et al., 2008). The particles obtained are monodisperse, with dimensions smaller than 100 nm, coated with silica shell using cetyltrimethylammonium bromide as surfactant for the transfer of hydrophobic nanocrystals to aqueous media and also as organic template in the sol-gel reaction. The silica- Fe_3O_4 particles were functionalized with PEG for further biomedical applications in cancer diagnosis and therapy.

Magnetite particles (nanoparticles with 5–300 nm or microparticles with 300–5,000 nm) have been surface functionalized and used as biosensors to recognize specific molecular targets. Three types of biosensors, (i) magnetic relaxation switches, (ii) magnetic particle relaxation sensors, and (iii) magnetoresistive sensors, were coated with different biosensing principles and magnetic materials for instrumentation (Josephson, 2009). Magnetic relaxation switch assay-sensors are based on the effects of the magnetic particles exert on water proton relaxation rates. Magnetic particle relaxation sensors determine the relaxation of the magnetic moment within the magnetic particle. Magnetoresistive sensors detect the presence of magnetic particles on the surface of electronic devices and are sensitive to changes in magnetic fields on their surface.

Magnetic resonance imaging (MRI) is a non-invasive technique without radiations exposure that can provide cross-sectional images from inside the solid materials and living organisms (Kim et al., 2003, Richardson et al., 2005). Magnetite nanoparticles are suitable for MRI contrast (Bean et al., 1959, Koenig et al., 1987). Magnetite particles have been proposed for oral use as magnetic resonance contrast agents and magnetic markers to study the gastrointestinal motility (Briggs et al., 1997, Ferreira et al., 2004). The gastric secretions include pepsin, mucus, and HCl (Paulev, 1999–2000) and magnetite particle dissolution may take place during their passing through the stomach. This could reduce the signal for MRI or for monitoring gastrointestinal motility. The problem that appears in this domain is to protect compounds from gastric environment and the xylan is a good candidate for this issue. Xylans are polysaccharides made from units of xylose and can be found as structural components in the cell walls of some green algae. Xylan-coated

magnetic microparticles were obtained (Andriola Silva et al., 2007) to protect magnetite from gastric dissolution.

5.2 Applications in catalysis

The aim of the catalysis is to search new catalysts that can be recovered when their activity is ended (Sankaranarayanapillai et al., 2010). The magnetic dispersions are attractive in separation applications as they offer high surface area and can be functionalized to apply to different molecular or cellular species. The magnetic fluids can be used as magnetically separable nanocatalytic systems that combine the advantages of homogeneous and heterogeneous catalysis (Lu et al., 2004). Novel thermoreversible magnetic fluids based on magnetite (Fe_3O_4) coated with a covalently anchored polymeric shell of poly(2-methoxyethyl methacrylate) (PMEMA) were synthesized by surface-initiated ATRP (Gelbrich et al., 2006). The coated particles form stable dispersions in methanol at temperatures above an upper critical solution temperature.

Magnetically recoverable heterogenized nanoparticle supported chiral Ru complexes were obtained and used in highly enantioselective asymmetric hydrogenation of aromatic ketones (Hu et al., 2005). The catalysts can be recycled by magnetic decantation and used for asymmetric hydrogenation for up to 14 times without loss of activity and enantioselectivity.

5.3 Industrial applications

Ferrofluids have numerous applications in rotary and linear seals as magneto transformers, sensors and pressure transducers (flow speed), inertial sensors (acceleration, inclination, and gravity) (Vekas, 2008), passive and active bearings, vibration dampers, linear and rotary drives, and chemical industry (microtechnologies) as chips, etc. Among the most important and commercial applications are developed magnetofluids rotating seals, which are found today in a variety of equipments, in electronics, nuclear industry, biotechnology, aeronautics and space applications.

Magnetofluids sealing advantages compared with mechanical seals are: sealing without leaking, life time (~ 5 years), only viscous friction, zero contamination, field operation (high vacuum (10^{-8} mbar) at approx. 10 bar), relatively simple construction and low manufacturing cost (www.roseal.topnet.ro).

For seals in dynamic regime, ferrofluids must obey a series of requirements (www.roseal.topnet.ro): high saturation magnetization (up to 50 kA/m (approx. 600 g)), physical properties adapted to operating conditions, especially in gas pressure and temperature sealed, high colloidal stability in intense and powerful magnetic field, Newtonian flow properties even in intense magnetic field.

Ferrofluids should operate at 150°C in continuous system or at temperatures of 200°C in intermittent system. They should also work in winter conditions (-20°C) or in space environment (-55°C) and to resist at nuclear radiation.

Researchers at NASA (Rosensweig et al., 1985) experienced ferrofluids in a spacecraft control system. While ferrofluids are used often in commercial processes, for the manufacture of CDs to create sophisticated suspension systems for cars, a researcher at

NASA (Kodama) (<http://www.interactivearchitecture.org/ferrofluid-sculptures-by-sachiko-kodama.html>) choose to use the dynamic qualities of these fluids. Thus, Kodama models their shape using a computer to control the electro-magnetic fields and creating different forms that continually reinvent themselves without the help of video effect. In the printing industry, special inks are used to protect valuable documents, labels, packaging or brand. Magnetic ink contains a pigment that produces magnetic pulses detected by magnetic reading devices, and messages can be read only by exposure to the equipment (<http://www.pricon.ro/ro/special.php>).

6. XPS results for core-shell magnetic nanoparticles

Magnetite particles coated by different Si-containing compounds (monomers and polymers) were prepared using co-precipitation method or a free-solvent synthesis. The covered magnetite particles were obtained in one step by co-precipitation of iron chloride salts in presence of monomers or (co)polymers adequately functionalized, as surfactants. When the co-precipitation was in the absence of surfactant, the covered particles were obtained in two steps, the first step consisting in the obtaining of magnetite particles and in the second step, the magnetite particles being covered with an adequate monomer or (co)polymer, specific for the desired application. The magnetite particles covered with aminosilane were obtained by replacement of the shell of magnetite-oleic acid particles which were synthesised by a free-solvent method. The average diameters of magnetic nanoparticles are depending on the synthesis method, are less than 1 μm and the smallest dimensions are obtained by free-solvent method (Table 2).

In this chapter we focused on the characterization of core-shell magnetic nanoparticles as determined by X-ray photoelectron spectroscopy (XPS). XPS, also known as Electron Spectroscopy for Chemical Analysis (ESCA), is an elemental analysis technique and used to determine quantitatively the atomic composition and surface chemistry. XPS spectra are obtained by irradiating a material with a beam of X-rays while simultaneously measuring the kinetic energy and number of electrons that escape from the top 1 to 10 nm of the material being analysed (Watts et al., 2003; http://en.wikipedia.org/wiki/X-ray_photoelectron_spectroscopy).

The surface of the magnetite particles modified with aminosilane (Ma-APTES) or with polydimethylsiloxane-carboxy-terminated poly(ethylene oxide) graft copolymer (Ma-PDMSgPEO-COOH) was carefully followed using XPS data.

Hydrophilic magnetite particles (Ma-APTES) were prepared by two methods:

- a. Co-precipitation of Fe^{2+} and Fe^{3+} ions in presence of ammoniacal solution according to the Massart method (Massart, 1981). The organic-inorganic “core-shell” magnetic particles (core: magnetite, shell: aminosilane monomer) were obtained by the condensation reaction between the hydroxyl groups on the magnetite surface and the ethoxyl groups from silane structure (Figure 6) (Durdureanu-Angheluta et al., 2008);
- b. The replacing, with aminosilane, of oleic acid of magnetite-oleic acid particles which were obtained by free-solvent method (Figure 7, Table 2) (Durdureanu-Angheluta et al., 2011b).

DLS, SEM and AFM analysis indicated the formation of low polydispersity particles, with a spherical morphology and dimensions of nanometers (Table 2).

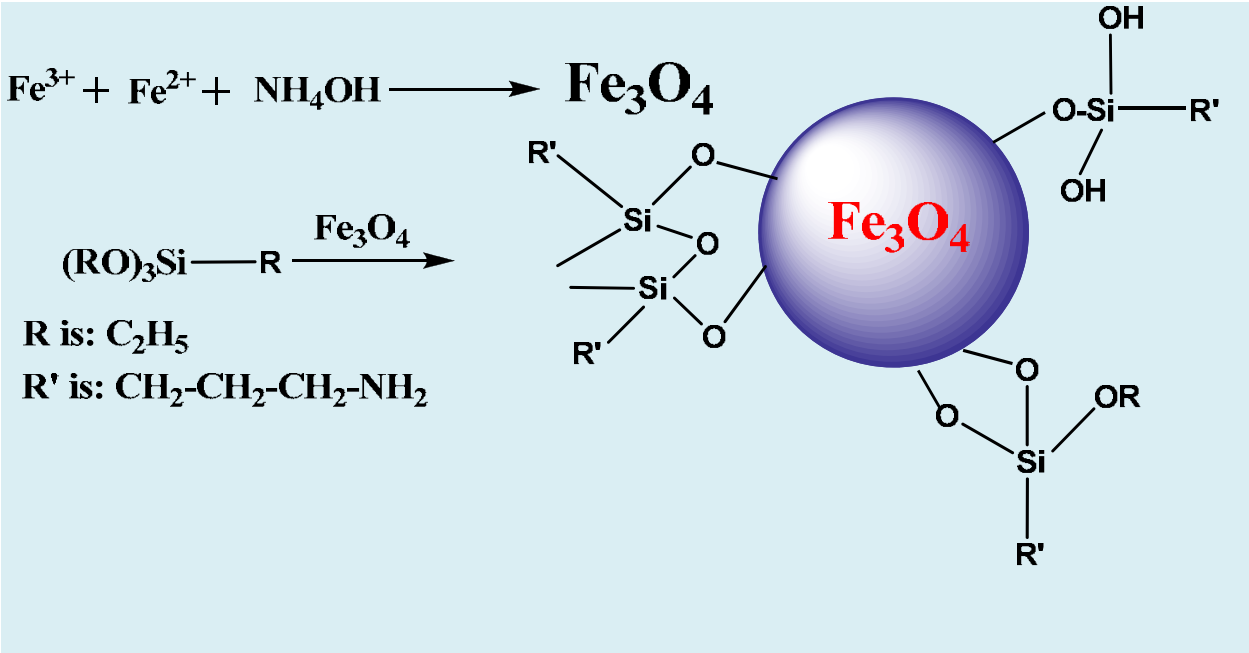


Fig. 6. Schematic principle of covalent bonding between magnetite core and ethoxyl groups of silane monomer (Durdureanu-Angheluta et al., 2008).

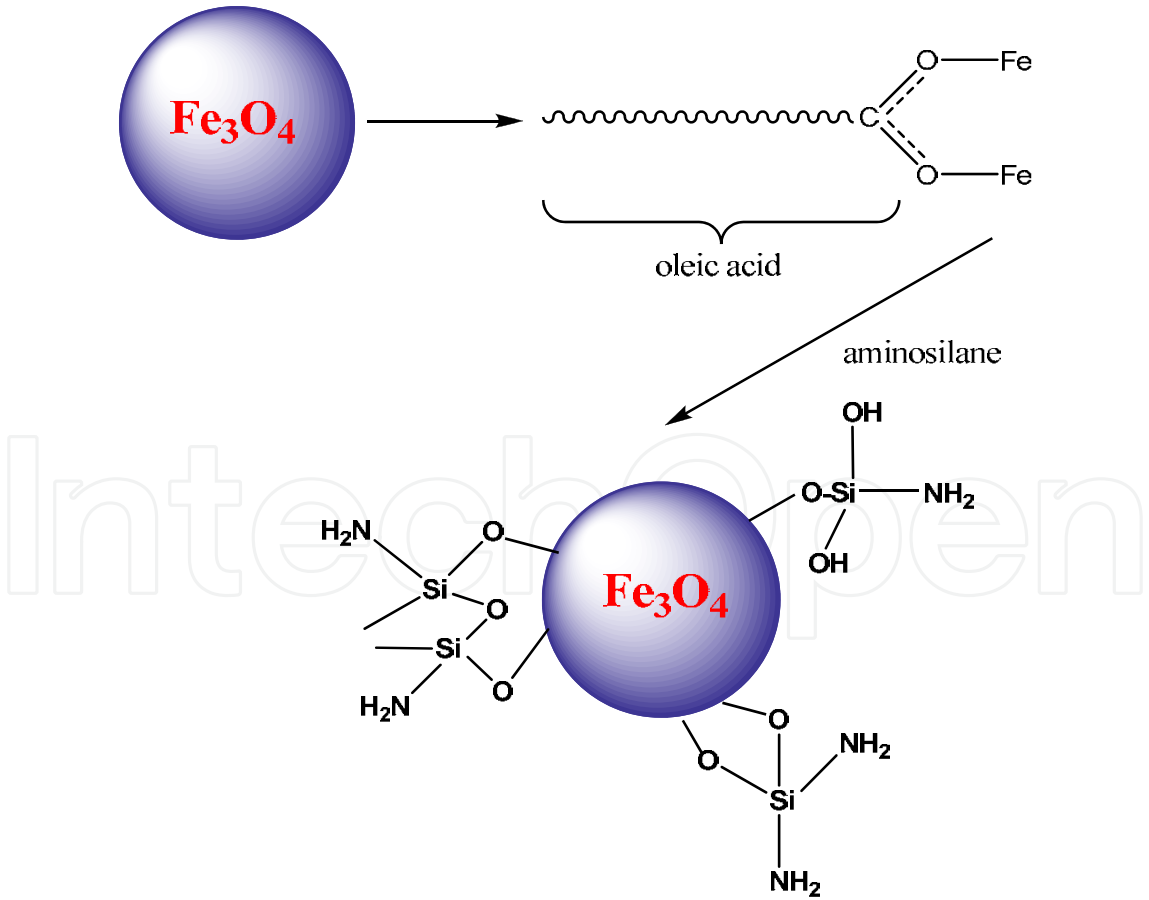


Fig. 7. Schematic representation of replacement of oleic acid shell with aminosilane monomer from magnetite-oleic acid particles (Durdureanu-Angheluta et al., 2011b).

Code sample	Shell type*	Fe ²⁺ /Fe ³⁺ **	Average diameter (nm) (DLS/AFM/SEM)	Magnetite synthesis method	Reference
Ma-ATES	allyltriethoxysilane	2	496	Co-precipitation	Durdureanu-Angheluta et al., 2008
Ma-MTES	triethoxymethylsilane	2	532	Co-precipitation	Durdureanu-Angheluta et al., 2008
Ma-APTES ₁	(3-aminopropyl)triethoxysilane	2	500	Co-precipitation	Durdureanu-Angheluta et al., 2008
Ma-GOPS	(3-glycidyloxypropyl)trimethoxysilane	2	270	Co-precipitation	Durdureanu-Angheluta et al., 2009
Ma-PDMS-TES	α-triethoxysilil-polydimethylsiloxane	0.5 1	670-800 200-400	One-step co-precipitation Two-step co-precipitation	Durdureanu-Angheluta et al., 2010
Ma-PDMSgE	siloxane functionalized with ester groups	2	173	Co-precipitation	Durdureanu-Angheluta et al., 2011a
Ma-PDMSgPEO-COOH	polydimethylsiloxane-carboxy terminated poly(ethylene oxide) graft copolymer	0.5	500	Co-precipitation	Pricop et al., 2010
Ma-OA	oleic acid	0.15 0.25 0.35	9 15 400	Free solvent synthesis	Durdureanu-Angheluta et al., 2011b
Ma-APTES ₂	(3-aminopropyl)triethoxysilane	0.15	33	Interchange of shell of Ma-OA	Durdureanu-Angheluta et al., 2011b

* Core of particles: magnetite (Ma)

** Fe²⁺/Fe³⁺: molar ratio used in the synthesis process

Table 2. Coated magnetite particles.

The XPS spectra for Ma-APTES₁ (Table 2 and Figure 6) were recorded on a XR6 monochromated X-ray source (Thermo Scientific Escalab250Xi), with AlKalpha radiation and a variable 200-900 μm spot size. Charge correction was performed using C1s at 284.8 eV as a reference. The presence of C 1s, O 1s, Si 2p, N 1s, as well Fe 2p peaks in XPS spectra on Ma-APTES₁ sample give a proof for the chemical structure of aminopropyltrisiloxy coated magnetite particles. The Si 2p spectrum (Figure 8) has a peak at 102.4 eV, which is typical of silicone atoms, but is also consistent with the oxysilane group expected in the sample.

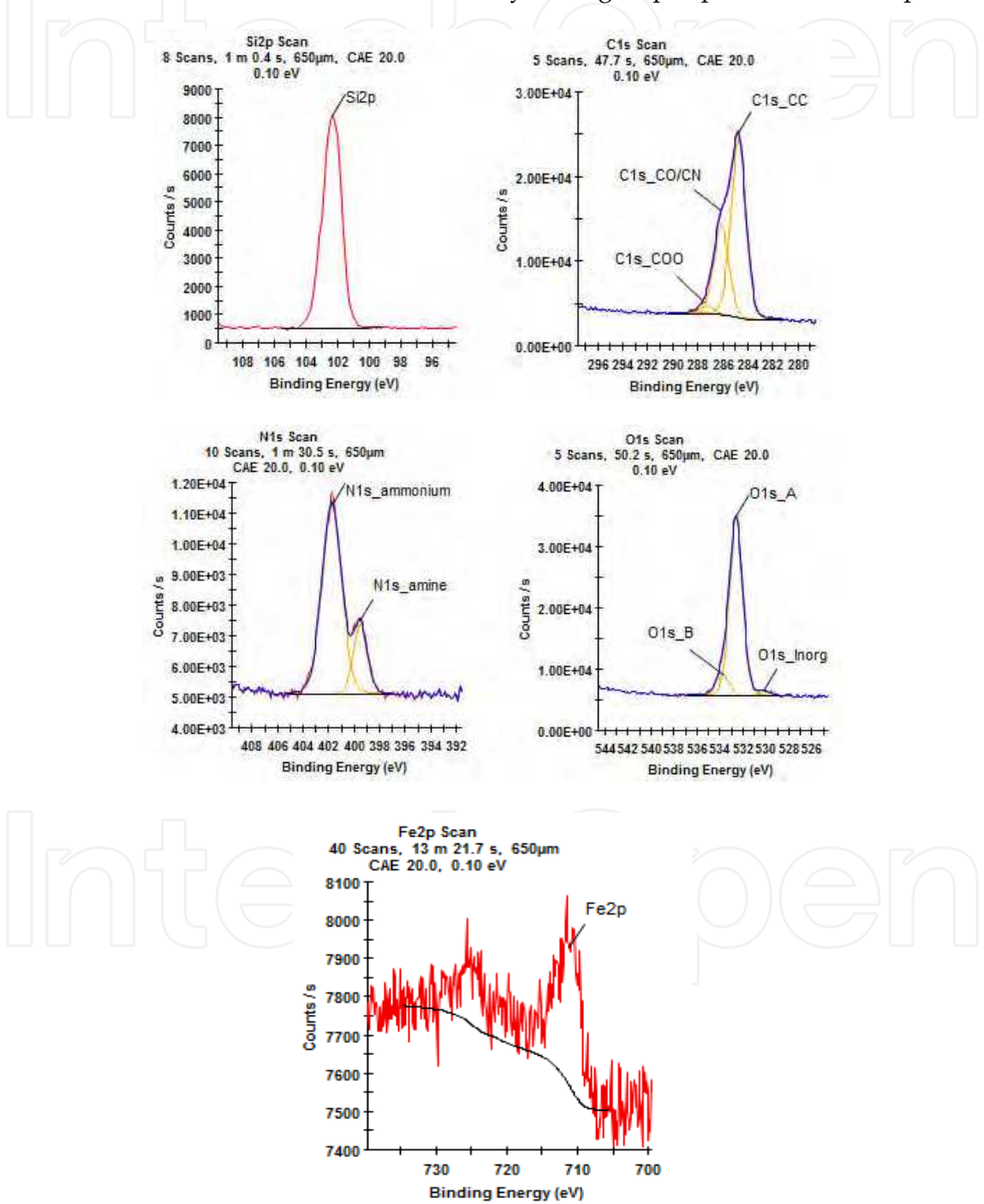


Fig. 8. High-resolution Si 2p, C 1p, N 1s, O 1s and Fe 2p XPS spectra for Ma-APTES₁ from Table 2.

C 1s (Figure 8) has two clear peaks, and requires three for a good fit. These have been assigned to C-C, C-O/C-N (should really be at least 2 peaks) and a weak peak tentatively assigned to O-C=O. N 1s (Figure 8) has two peaks, which appear to represent amine and ammonium (or ammonia) in the sample. O 1s (Figure 8) has three peaks, probably from different C-O and Si-O states, and also an inorganic oxide which is presumably the magnetite. Fe 2p (Figure 8) is extremely weak, so it is difficult to confirm the iron chemistry in more detail than "oxide".

The structure of Ma-APTES₂ coated magnetite nanoparticles (Table 2) was demonstrated by X-ray photoelectron spectroscopy (XPS), showing the formation of chemical bonds between the surface of magnetite particles and the oxygen atoms of the shells (Durdureanu-Angheluta et al., 2011b). XPS was performed on a KRATOS Axis Nova (Kratos Analytical, Manchester, United Kingdom), using AlK α radiation, with 20 mA current and 15 kV voltage (300 W), and base pressure of 10⁻⁸ to 10⁻⁹ Torr in the sample chamber. The incident monochromated X-ray beam was focused on a 0.7 mm x 0.3 mm area of the surface. The XPS survey spectra for the magnetite sample was collected in the range of -5÷1200 eV with a resolution of 1 eV and a pass energy of 160 eV. The high resolution spectra for all the elements identified from the survey spectra were collected using pass energy of 20 eV and a step size of 0.1 eV. The data were analyzed using the Vision Processing software (Vision2 software, Version 2.2.8). A linear background was subtracted before the peak areas were corrected. The binding energy of the C 1s peak was normalized to 285 eV.

In Figure 9 the peak at 283.4 eV belonging to the carbon from C-Si, 285.2 eV, was assigned to the carbon in the aliphatic chain (C-C) and the peak at 286.6 eV was ascribed to the carbon from C-N. The others two small peaks at 287.9 and 289.2 eV belonging to the carboxylate (C=O, COO⁻), were in agreement with the data obtained in the previous literature (Zhang et al., 2006), and come from traces of oleic acid. The lack of peak at 290 eV indicates the absence of free oleic acid. The bonding energies at 102.3 and 103.0 eV (Figure 10) were the characteristic peaks from Si-O and Si-C bonds, respectively. Deconvolution of the N 2p peaks (Figure 11) indicated, at 399.3 and 400.8 eV, the presence of amino groups (NH₂) and tertiary amino groups, respectively. From the deconvolution of O 1s peaks (Figure 12) resulted three peaks from magnetite (around 530.8 eV), HO-Fe (around 531.4 eV) (Khurshid et al., 2009) and O-Si oxygen bonds (around 532.4 eV). The peak at around 535 eV could be due to the traces of oleic acid in the layer. The bonding energies at 710.6 and 723.7 eV were the characteristic peaks from Fe 2p_{3/2} and Fe 2p_{1/2}, respectively. Deconvolution of the Fe 2p has shown the presence of Fe³⁺ and Fe²⁺ in both Fe 2p_{2/3} and Fe 2p_{1/2} regions (Fe³⁺ 2p_{2/3}/Fe³⁺ 2p_{1/2}: 710.7 eV/724.4 eV; Fe²⁺ 2p_{2/3}/Fe²⁺ 2p_{1/2}: 709.2 eV/722 eV). It should be mention that an energy difference of 13.1 eV between 2p_{3/2} and 2p_{1/2} peaks indicates that the material has, as dominant phase, Fe₃O₄ and, as secondary phase, siloxy-Fe and carboxylate-Fe bonds (Zhang et al., 2006). Also, XPS results on the Fe²⁺/Fe³⁺ molar ratio of 0.13 (Figure 13) confirm the Fe²⁺/Fe³⁺ molar ratio used in the synthesis of the magnetite particles (Ma-OA from Table 2).

Hydrophobic magnetite particles (Ma-PDMSgPEO-COOH) were obtained by co-precipitation of iron salts in water at pH around 11 in the presence of dichloromethane solution of siloxane copolymer (PDMSgPEO-COOH) (Figure 10) (Pricop et al., 2010).

Dimensional analysis of magnetite particles coated with PDMSgPEO-COOH (Table 2) showed core-shell morphology of an approximate spherical shape, with an average

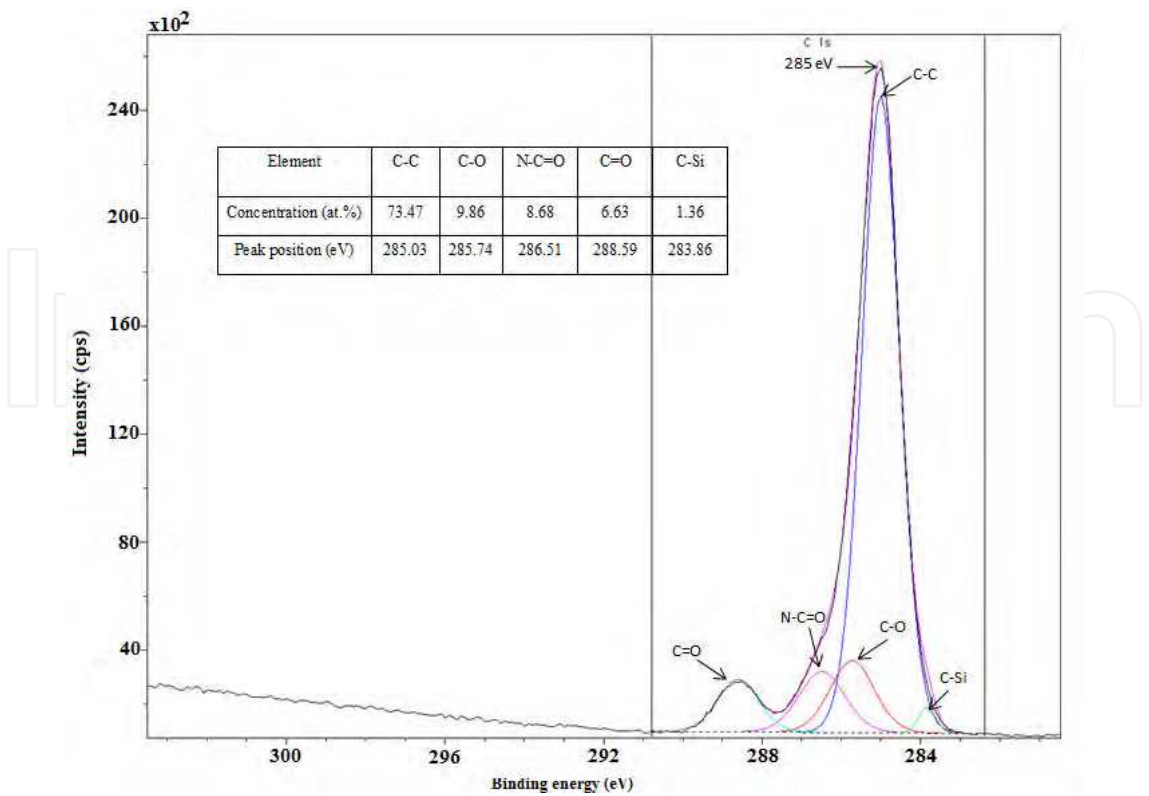


Fig. 9. High-resolution C 1s XPS spectra for Ma-APTES₂ from Table 2 (Durdureanu-Angheluta et al., 2011b).

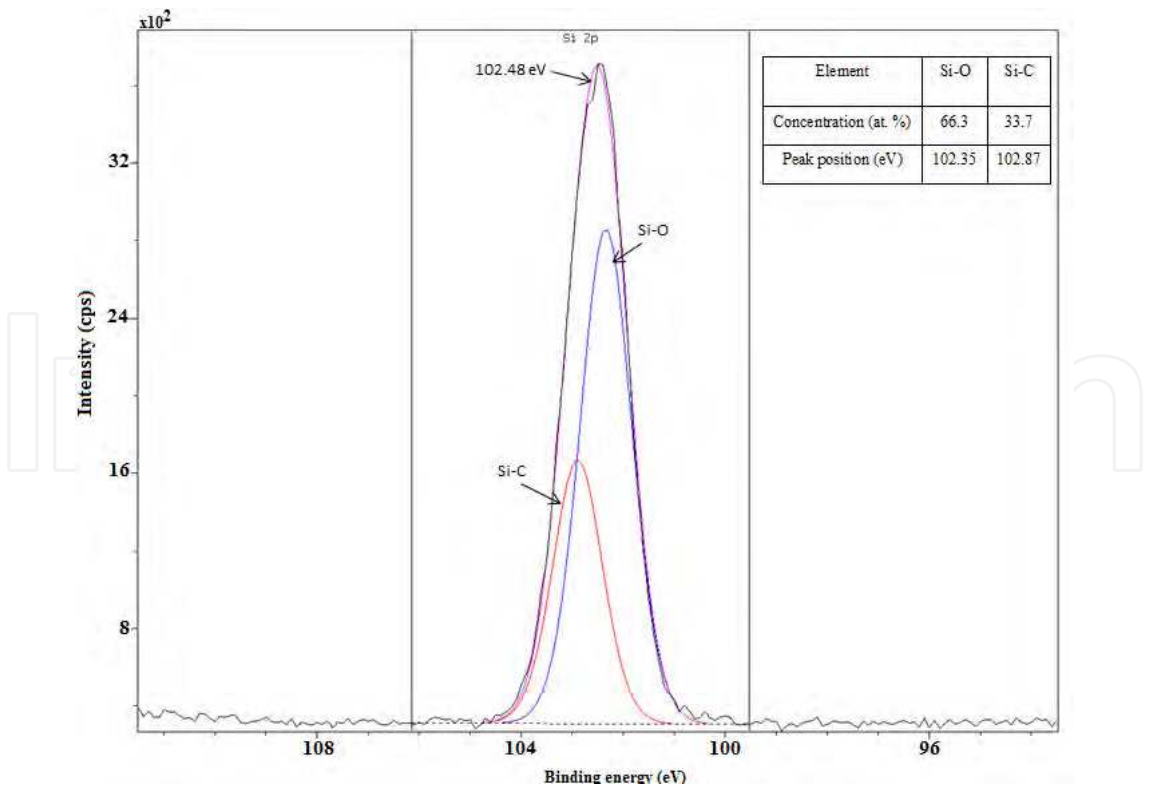


Fig. 10. High-resolution Si 2p XPS spectra for Ma-APTES₂ from Table 2 (Durdureanu-Angheluta et al., 2011b).

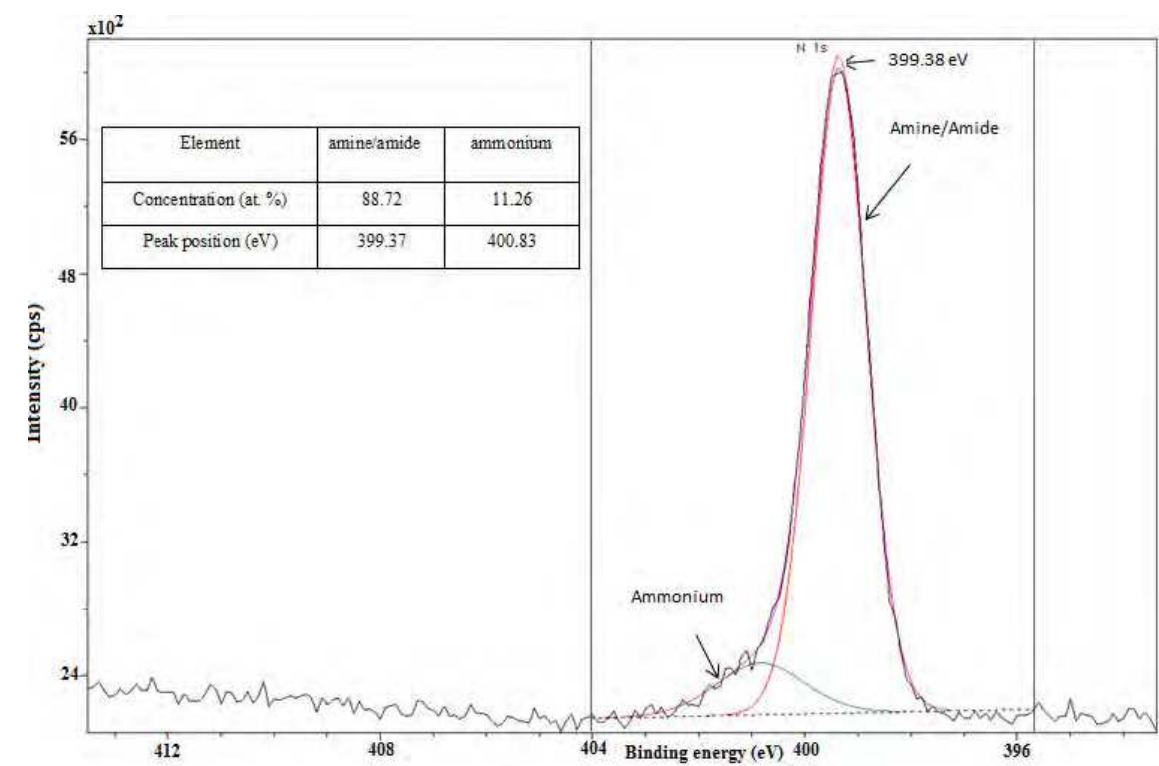


Fig. 11. High-resolution N 1s XPS spectra for Ma-APTES₂ from Table 2 (Durdureanu-Angheluta et al., 2011b).

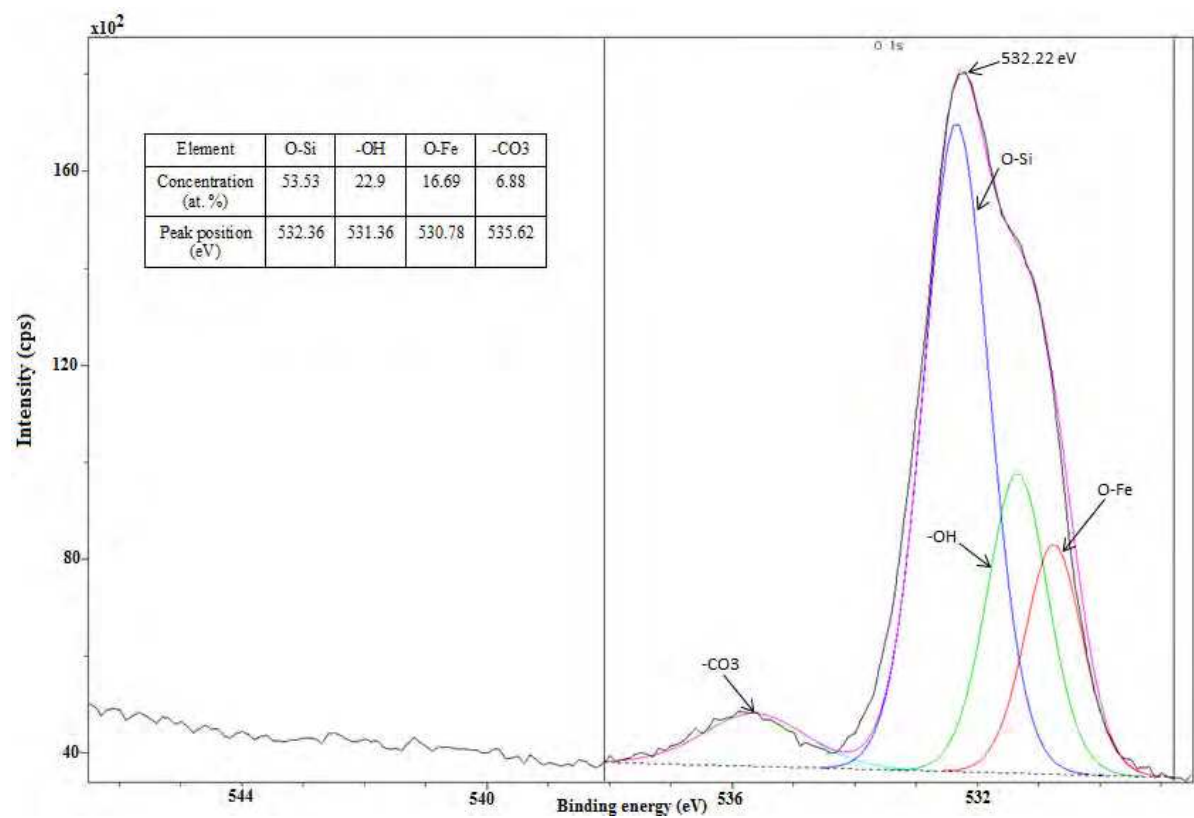


Fig. 12. High-resolution O 1s XPS spectra for Ma-APTES₂ from Table 2 (Durdureanu-Angheluta et al., 2011b)

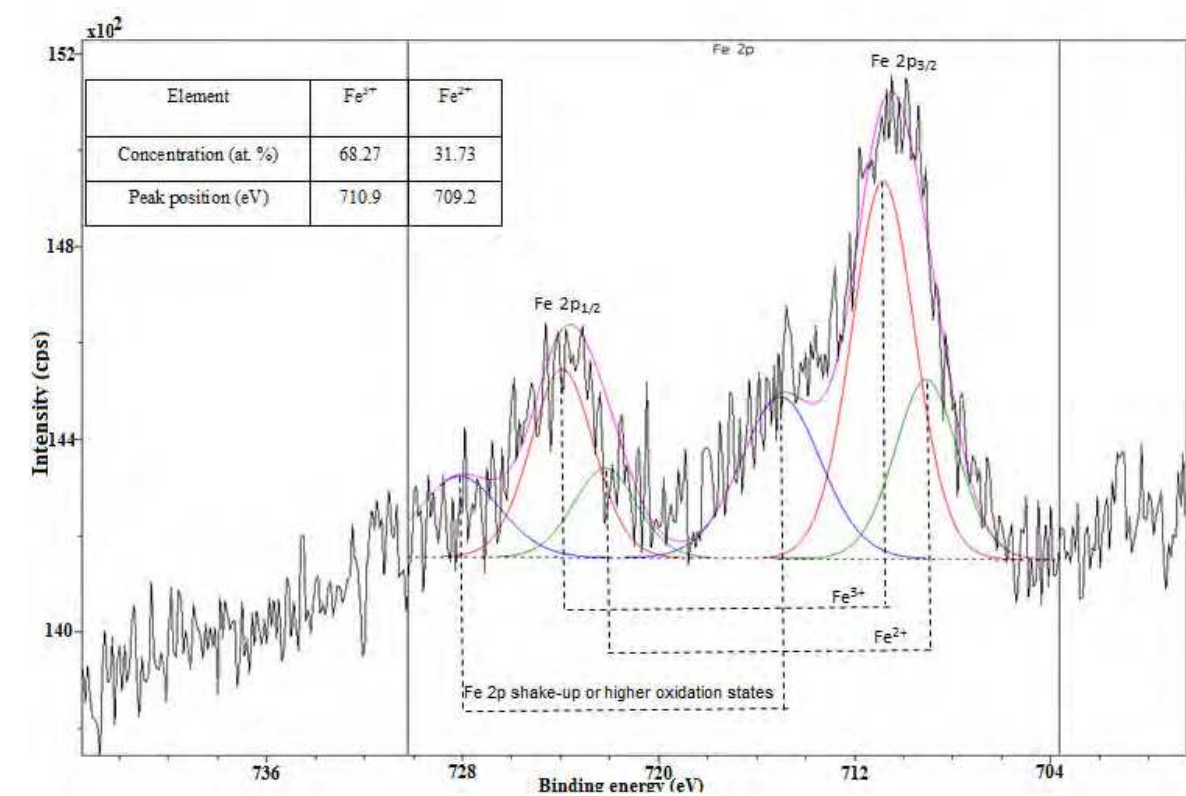


Fig. 13. High-resolution Fe 2p XPS spectra for Ma-APTES₂ from Table 2 (Durdureanu-Angheluta et al., 2011b)

diameter of 500 nm. The amphiphilic graft copolymer was proved to be efficient as surfactant in the preparation of magnetite particles with a hydrophobic siloxane shell (Pricop et al., 2010).

Ma-PDMSgPEO-COOH particles were analysed on the AXIS Nova X-ray photoelectron spectrometer built around the AXIS technology. Data reduction and processing was performed using Kratos' Vision 2 Processing software.

XPS scans of Si 2p atoms evidenced the appearance of the peaks at around 100 and 102 eV, characteristic for Si-O and Si-C bonds, respectively (Figure 15). C 1s atoms present specific peaks for CH₃-Si, CH₂-Si, C-O-C, C-C, C=O, O-C=O (Figure 15) in the 282 to 289 eV interval. The deconvolution of O 1s shows the presence of the peak at around 530 eV (Figure 15), corresponding to the metallic oxide and the presence of two peaks, with higher binding energies due to hydroxyl groups or from the coating of the sample. The deconvolution of Fe 2p atoms (Figure 15) presents two peaks at 710.7 and 712.8 eV, attributed to Fe³⁺ 2p_{2/3} and Fe²⁺ 2p_{2/3}, respectively.

The presence of peaks at 724.0 and 726.0 eV of Fe³⁺ 2p_{1/2} and Fe²⁺ 2p_{1/2}, respectively and the difference of 13.2 eV between Fe 2p_{3/2} and Fe 2p_{1/2} explain the presence of the carboxylate-Fe bonds (Zhang et. al., 2006).

It should be underlined that the XPS results were substantiated by FT-IR data, indicating the formation of the hydrogen or covalent bonds between the iron oxide substrate and the functional group of the surfactant.

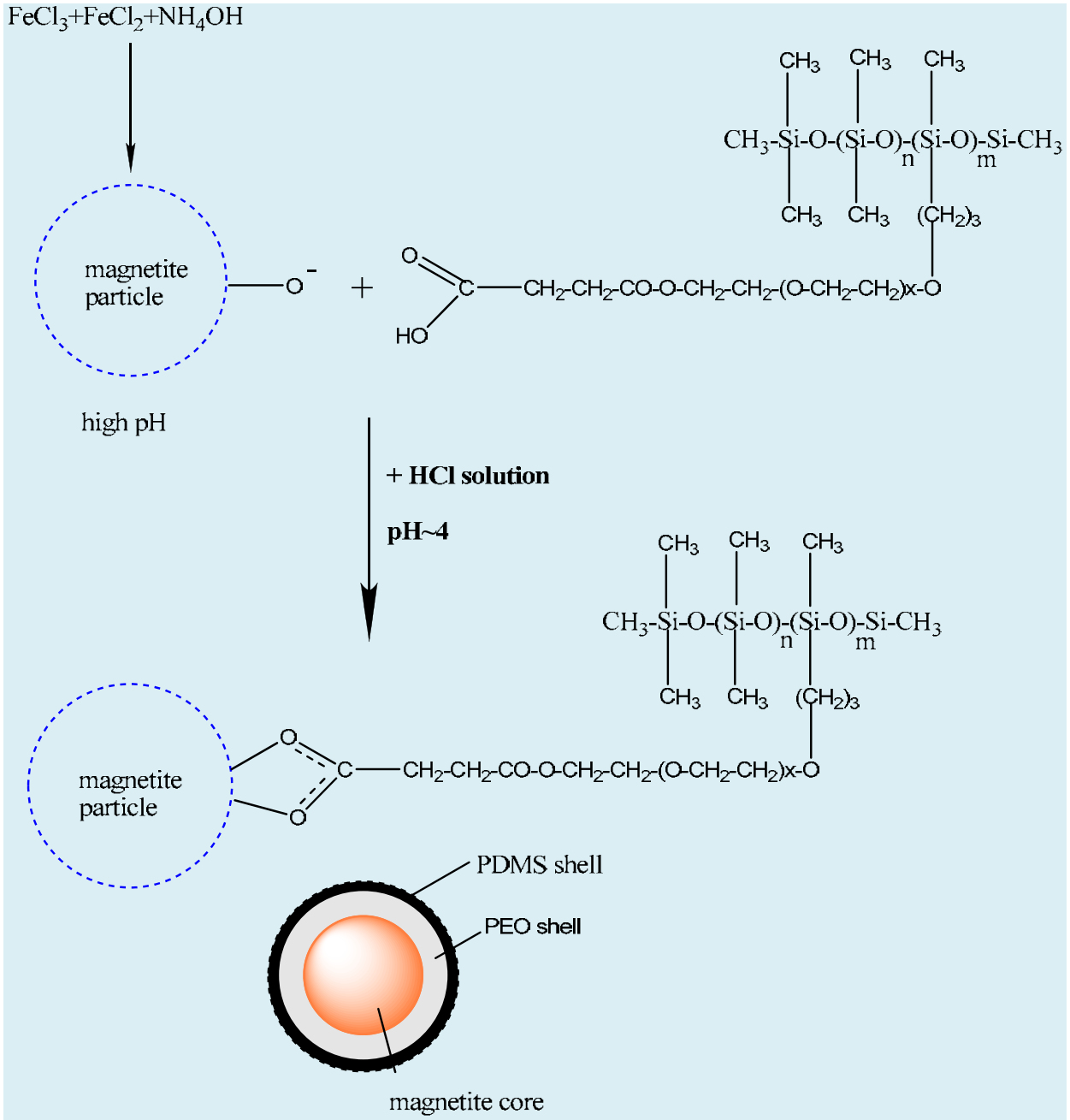


Fig. 14. Schematic principle of hydrogen bonding between magnetic core and the hydrophobic siloxane shell.

The core-shell magnetic particles present superparamagnetic properties and their saturation magnetization values are correlated with those reported by the literature.

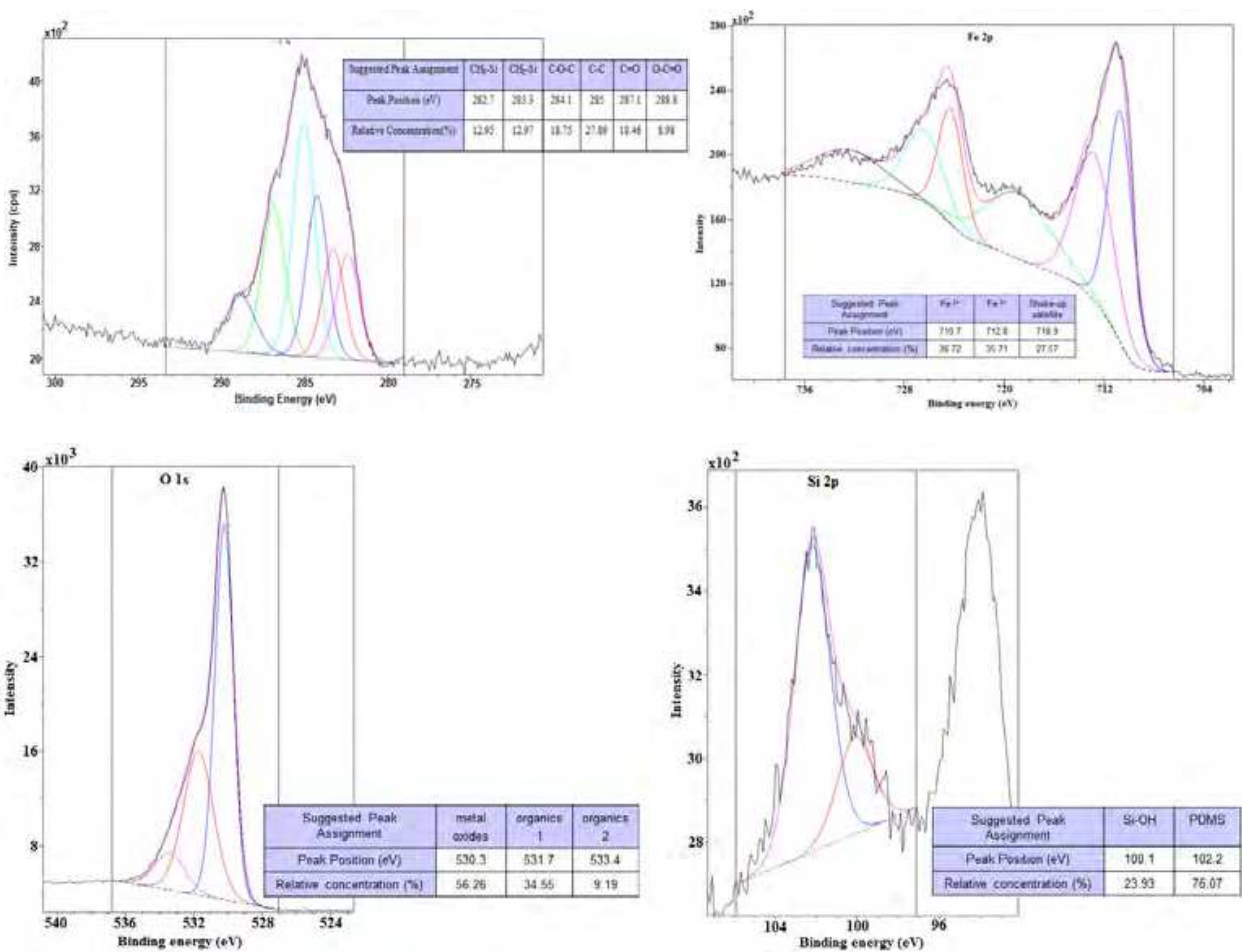


Fig. 15. High-resolution C 1s, Fe 2p, O 1s and Si 2p spectra for Ma-PDMSgPEO-COOH from Table 2.

7. Conclusion

Many studies on magnetite particles and their applications in various fields were published. This chapter has embedded a few examples, also including our own studies on modified magnetite particles with different fictionalizations, specific for the desired application. We emphasized the two different methods that can be employed for core-shell particles preparation. The results obtained by our group point that free-solvent technique is the more versatile one, allowing to obtain particles with smaller sizes and higher stability in magnetic fluids, important characteristics for biomedical applications. These materials are very promising for the immobilization of biologically active compounds.

8. Acknowledgment

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Materials are important to mankind because of the benefits that can be derived from the manipulation of their properties, for example electrical conductivity, dielectric constant, magnetization, optical transmittance, strength and toughness. Materials science is a broad field and can be considered to be an interdisciplinary area. Included within it are the studies of the structure and properties of any material, the creation of new types of materials, and the manipulation of a material's properties to suit the needs of a specific application. The contributors of the chapters in this book have various areas of expertise. therefore this book is interdisciplinary and is written for readers with backgrounds in physical science. The book consists of fourteen chapters that have been divided into four sections. Section one includes five chapters on advanced materials and processing. Section two includes two chapters on bio-materials which deal with the preparation and modification of new types of bio-materials. Section three consists of three chapters on nanomaterials, specifically the study of carbon nanotubes, nano-machining, and nanoparticles. Section four includes four chapters on optical materials.

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