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Magnetically Responsive Activated Carbons for Bio - and Environmental Applications

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Abstract – *Activated carbons are very important materials used e.g. as adsorbents, catalysts and catalyst supports. The application potential of activated carbons can be increased by their magnetic modification. Magnetically responsive activated carbons can be easily separated from complex systems or targeted to a desired place using an external magnetic field. This short review summarizes typical procedures used to convert non-magnetic activated carbons into their magnetic derivatives, and typical applications of magnetic activated carbons in biosciences, biotechnology, medicine, analytical chemistry and environmental technology.* ***Copyright © 2012 Praise Worthy Prize S.r.l. - All rights reserved.***

***Keywords**: Activated Carbon, Magnetic Modification, Magnetic Separation*

I. Introduction

Activated carbons (AC), also known as activated charcoal, are very important and versatile materials used e.g. as effective adsorbents, catalysts and catalyst supports exhibiting large surface area, highly developed porosity, variable characteristics of surface chemistry, and high degree of surface reactivity. Activated carbons are used for different purposes such as the removal of pollutants from gaseous or liquid phases and the purification or recovery of chemicals. Activated carbon adsorption is of interest to many economic sectors and concern areas and industries as diverse as food, pharmaceutical, chemical, petroleum, nuclear or automobile, as well as for the treatment of drinking water, industrial and urban waste water, etc. Unfortunately, due to their high production costs, these materials tend to be more expensive than other adsorbents [1].

Activated carbons can be prepared from different plant materials. AC production costs can be reduced by choosing a cheap raw material or by applying a proper production method, such as low temperature processing. The use of waste materials for the preparation of AC is also very attractive from the point of view of their contribution to decrease the costs of waste disposal, therefore helping environmental protection [1].

There are several possible approaches how to improve the application potential of activated carbons. One of them is the formation of composite materials prepared by incorporation of appropriate inorganic (nano) particles in the structure of activated carbons. In this short review we will focus on magnetic derivatives of activated carbons (MAC) which can be easily separated from complex systems or targeted to a desired place using an external magnetic field.

II. Magnetic Modification of Activated Carbon

Magnetic nano- and microparticles have found many interesting applications in various areas of biosciences, medicine, biotechnology and environmental technology, electronics, nondestructive testing etc. [2], [3]. Different types of responses of such materials to external magnetic field enable various applications, namely selective separation, targeting and localization of magnetically responsive nano- and microparticles and other relevant materials using an external magnetic field (e.g. using an appropriate magnetic separator, permanent magnet or electromagnet), heat generation (which is caused by magnetic particles subjected to high frequency alternating magnetic field), increase of a negative T_2 contrast by magnetic iron oxides nanoparticles during magnetic resonance imaging or great increase of apparent viscosity of magnetorheological fluids when subjected to a magnetic field. In addition, magnetic nano- and microparticles can be used for magnetic modification of diamagnetic biological materials (e.g. prokaryotic and eukaryotic cells or plant-derived materials), biopolymers, organic polymers and inorganic materials and for magnetic labeling of biologically active compounds and affinity ligands (e.g. antibodies, enzymes, aptamers etc.) [4], [5]. Many procedures for the conversion of non-magnetic activated carbon particles into their magnetic derivatives have been described. Magnetic modification is usually caused by the presence of magnetic nano- or microparticles within the carbon particles pores, on the carbon particles surface or within the gels containing co-entrapped charcoal particles. Magnetic nano- and microparticles modifying activated carbon are mainly iron oxides magnetite or maghemite, or different types of ferrites. However, in

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some cases also metallic iron and nickel have been synthesized and used as magnetic labels.

The simplest and most often used approach for magnetic modification of AC is based on the alkaline precipitation of ferrous and ferric salts in the presence of activated carbon, followed by heating of the aqueous suspension. Magnetic iron oxides (magnetite, maghemite or their mixtures) or different types of ferrites are usually formed. Typical examples of described procedures employing precipitation reactions are shown in Table I.

TABLE I
CONVERSION OF ACTIVATED CARBONS (CHARCOAL) INTO THEIR MAGNETIC DERIVATIVES USING CHEMICAL PRECIPITATION PROCEDURES

Modification Procedure	References
Precipitation of magnetite from $FeSO_4$ and $Fe_2(SO_4)_3$ by NaOH in the presence of charcoal, followed by aging for 24 h and heating at 473 K	[6]
Precipitation of iron oxides from $FeSO_4$ and $FeCl_3$ by NaOH in the presence of charcoal, followed by drying at 100 °C for 3 h	[7]
Precipitation of hydrated iron oxides from $FeSO_4$ by NaOH in the presence of charcoal, followed by heating to 100 °C for 1 h	[8]
Activated carbon was suspended in NaOH solution and heated to 100 °C; then a solution of $Fe(NO_3)_3$ and $Co(NO_3)_2$ was quickly poured into the AC suspension and refluxed at 100 °C for 2 h	[9]
Bamboo charcoal powder was suspended in $Fe(NO_3)_3$, $Zn(NO_3)_2$, $Ni(NO_3)_2$ and aqueous ammonia solution and then heated in an autoclave at 180 °C for 2 h and air cooled to room temperature	[10]
Activated carbon was suspended in $CuCl_2$ and $FeCl_3$ solution, followed by NaOH solution addition and heating to 98-100 °C for 2 h	[11]
$FeCl_3$ and $FeSO_4$ solution was mixed with NaOH solution to keep pH value of 9.5, then activated carbon was added and the obtained material was dried in an oven at 100 °C for 3 h	[12]

Another large group of methods for magnetic modifications is based on the treatment of activated carbon impregnated with iron or nickel salts by heating at high temperatures. Depending on the salt, heating conditions and atmosphere used various magnetic modifiers can be formed including magnetic iron oxides or nickel particles.

Typical examples of described procedures employing heating of impregnated activated carbons are shown in Table II.

In alternative procedures magnetic activated carbon was prepared by mixing carbon powder with the suspension of magnetic iron oxides prepared by standard precipitation procedure; the mixture was stirred and then dried at 40°C [22]. Also magnetic fluid stabilized with triethanolamine oleate was used for rapid preparation of MAC by simple impregnation procedure followed by washing and drying at 90°C in air [23]. In another described procedure activated carbon was mixed with iron powder followed by treatment in a high-energy planetary ball mill [24].

In another approach magnetite microparticles were coated with a thin layer of epoxy resin which subsequently enabled attachment of activated carbon [25].

TABLE II
CONVERSION OF ACTIVATED CARBON (CHARCOAL) INTO ITS MAGNETIC DERIVATIVE BY HIGH TEMPERATURE TREATMENT

Modification Procedure	References
Activated carbon was impregnated with an aqueous solution of sucrose and $Ni(NO_3)_2$, followed by heating at 600 °C under N_2 for 3 hours. Ni nanoparticles were formed within the porous AC matrix	[13]
A solution of $Ni(NO_3)_2$ was dropped into NaOH solution, then ethanol solution of phenolic resin was added followed by solvent evaporation at 333 K and carbonization under argon atmosphere at 873 K	[14]
Impregnation of activated carbon with $Fe(NO_3)_3$ solution followed by drying at 90 °C and heated to 700 °C under argon; then benzene vapor was introduced	[15]
Activated carbon from rice husk was modified with HNO_3 for 3 h at 80 °C followed by suspending in $Fe(NO_3)_3$ and drying. Thermal treatment was conducted at 750 °C for 3 h in the presence of N_2 to enable formation of magnetite nanoparticles	[16]
Dried chitosan microspheres were immersed in $(NH_4)_3[Fe(C_2O_4)_3]$ solution followed by washing and drying, then the sample was carbonized under Ar atmosphere at 700-1000 °C for 4 h	[17]
Activated carbon was suspended in $Fe(NO_3)_3$; after drying it was heated to 800 °C in N_2 atmosphere and after cooling heated at 850 °C in CO_2 atmosphere for 1.5 h	[18]
A mixture of the anthracite powder, coal tar, Ni(NO3)2 and water was mixed and extruded in the form of 1 cm cylinders. After drying the material was carbonized under a flow of N2 at 600 °C and then activated at 880 °C under a flow of N2	[19]
Activated carbon was impregnated with $Fe(NO_3)_3$ solution and then with ethylene glycol. The impregnated sample was subjected to heat treatment under N_2 atmosphere at a temperature 250-450 °C for 2 h	[20]
Activated carbon was filled with a $Fe(NO_3)_3$ solution in ethanol and then dried at 90 °C for 2 h. Then the sample was impregnated with ethylene glycol followed by heat treatment under N_2 atmosphere at a temperature 350 or 450 °C for 2 h	[21]

Encapsulation of activated carbon together with magnetic particles in an appropriate biopolymer or synthetic polymer gel is another possibility for MAC formation, as shown in Table III.

III. Bioapplications of Magnetically Responsive Activated Carbons

Magnetic activated carbons have been efficiently used for both biochemical assays and medical applications employing their ability to be selectively separated from the reaction mixture or targeted to the specific area. Radioimmunoassay (RIA) is a very sensitive in vitro assay technique used to measure concentrations of different antigens (for example, hormone levels in the blood) by use of antibodies. Radioimmunoassays require a separation step prior to isotope counting because it is impossible to distinguish between the radioactivity in the antibody-bound and –free fractions.

TABLE III
CONVERSION OF ACTIVATED CARBONS (CHARCOAL) INTO THEIR MAGNETIC DERIVATIVES BY ENCAPSULATION

Modification Procedure	Refe-rences
Activated carbon was mixed with alginate solution and citrate stabilized ferrofluid and then the suspension was added dropwise into a $CaCl_2$ solution	[26]
Cellulose was dissolved in a cooled NaOH/urea solution followed by the addition of maghemite nanoparticles and activated carbon; the suspension was added dropwise into a NaCl solution. The formed beads were cross-linked with epichlorohydrin	[27]
Charcoal and magnetisable ferric oxide were entrapped in a polyacrylamide gel followed by lyophilisation and micronisation	[28]
Charcoal and barium ferrite microparticles were mixed with bovine serum albumin solution followed by emulsification in n-butanol – castor oil – glutaraldehyde continuous phase	[29]
Charcoal and magnetisable ferric oxide were entrapped in a polyacrylamide gel followed by drying at 80 °C overnight and milling to obtain particles of less then 50 μm in diameter	[30]
Activated carbon was suspended in NaOH solution and heated to 100 °C; then a solution of $Fe(NO_3)_3$ and $Co(NO_3)_2$ was quickly poured into the AC suspension and refluxed at 100 °C for 2 h. This material was added to Na alginate solution followed by pouring dropwise into $CaCl_2$ solution	[31]

One of the possible techniques is the addition of appropriate adsorbent to adsorb the unbound analyte; both AC and MAC were successfully used for this purpose. Magnetic derivative enabled simple separation of the adsorbent with the bound free analyte using a magnetic separator [32]. Magnetically responsive activated carbon has been successfully used in radioimmunoassay of important analytes, such as steroids and small polypeptide hormones [30], digoxin [28], vitamin B12 [29] or progesterone [33]. Similar approach was used for rapid, high-throughput transglutaminase assay where magnetic dextran-coated charcoal has been used to capture the low-molecular-weight reagent from the reaction mixture [34].

Magnetite microparticles covered with activated carbon by using epoxy resin as an adhesive were used as a carrier for the immobilization of *Saccharomyces cerevisiae* cells. The immobilized cells were used in batch and continuous alcoholic fermentation. The adsorption of the yeast cells obeyed the Langmuir isotherm equation. Satisfactory results were obtained both in the case of simple adsorption and adsorption followed by glutaraldehyde cross-linking [25].

The same magnetic material was used to separate *Saccharomyces cerevisiae* cells from aqueous suspensions using magnetically stabilized fluidized beds (MSFB) that utilized a horizontal magnetic field; the effects of some parameters, such as bed porosity and height, liquid flow rate and inlet concentration on cell removal efficiency and breakthrough curves were studied [35].

Magnetic drug delivery has been an active field of study for several decades. Proposed in the 1970s, the concept of magnetic drug targeting is to inject a magnetically responsive material containing bound, adsorbed or entrapped drug and then to use an externally placed magnet or an advanced high field gradient magnet [36] to guide the magnetic drug to the targeted site.

Ferro-carbon particles, in which the Fe core acts as the magnetically susceptible component and activated carbon as the drug carrier, represent one type of important materials for drug targeting because the susceptibility of metallic iron is many times higher than that of magnetite. Activated carbon is a good choice as a coating, due to its high surface area and known adsorption–desorption properties for many molecules including peptides, proteins and drugs. The molecular adsorption to activated carbon depends on the carbon surface, pore size and the source of the material [37].

An antitumor drug paclitaxel was bound to ferro-carbon particles (0.5 – 2 μm in diameter) which could be localized quantitatively at capillary (0.2 cm s^{-1}) to arteriole (28 cm s^{-1}) flow rates under the effect of magnetic field. In blood serum, 38 % of adsorbed drug could be released from the carrier in 24 hours [38]. Similar carbon-based magnetic carrier also enabled efficient binding of doxorubicin, mitomycin C, camptothecin, methotrexate, verapamil and 9A. With the aid of an externally positioned permanent dipole magnet, the drug-loaded carrier could be localized and retained within a tumor mass [39].

A one-step radiolabeling procedure of magnetic activated carbon particles with the therapeutic β-emitter rhenium-188 has been developed and the prepared material was subsequently targeted to solid tumors; it enabled to deliver therapeutically relevant doses of radiation to tumors while minimizing radiation exposure to surrounding tissues or organs [40].

IV. Environmental Technology Applications of Magnetically Responsive Activated Carbons

Activated carbons have been often used as efficient adsorbents for many types of organic and inorganic xenobiotics, radionuclides, noble metals etc. Magnetically responsive carbon derivatives usually exhibit similar (or sometimes even better) adsorption of the target compounds as the native activated carbon and in addition, they can be easily separated. Recently, a large number of studies has appeared in the scientific literature describing the applications of various modifications of MAC for xenobiotics removal from contaminated water resources. MAC is a additional material to magnetically responsive biocomposites used for the same purpose [41]. Typical examples of described procedures employing MAC for the separation and/or removal of organic compounds are shown in Table IV, while Table V presents typical examples of inorganic compounds separations.

A novel magnetically separable composite photocatalyst, titania-coated magnetic activated carbon, was prepared by depositing of anatase titania onto the surface of magnetic activated carbon. The photocatalytic activity of the samples was determined by degradation of reactive brilliant red X-3B under either UV or visible irradiation; this activity was high and the composite photocatalyst could be reused with a little reduction of its photocatalytic activity [22].

Activated carbon modified with zero-valent iron deposits exhibited dehalogenation activity for chlorinated and brominated C_1 and C_2 hydrocarbons in aqueous solutions. The pollutants were collected and enriched at the MAC surface and destructed at the Fe clusters by reductive dechlorination. Lifetimes of the material in the order of several weeks have already been achieved in laboratory studies [42].

Regeneration of MAC after finishing an adsorption process is important in order to keep the process expenses as low as possible. Recently magnetic $CuFe_2O_4$ - activated carbon composite adsorbent has been prepared using a chemical co-precipitation method. After adsorption of acid orange II the composite regeneration was performed by heating in an inert atmosphere. The results indicated that the $CuFe_2O_4$ particles could effectively catalyze the thermal pyrolysis of the adsorbed dye. The results of regeneration tests suggested that almost all adsorption capacity of the composite adsorbent was re-established after thermal treatment and it could be reused for several cycles [11]. An alternative regeneration procedure was based on the use of hydrogen peroxide; the presence of the Fe_3O_4 nanoparticles in MAC was beneficial for achieving high regeneration efficiency [43].

Magnetic activated carbon has been used as a dyes adsorbent during the development of "magnetic solid-phase extraction" (MSPE); in this procedure magnetic adsorbent is added to a solution or suspension containing the target analyte. The analyte is adsorbed onto the magnetic adsorbent and then the adsorbent with adsorbed analyte is recovered from the suspension using an appropriate magnetic separator. The analyte is consequently eluted from the recovered adsorbent and analyzed. Up to 460-fold enrichment of analytes was observed using MAC as an adsorbent and water soluble dyes as analytes [49].

Magnetic activated carbon can also become an interesting, easy to prepare magnetic composite material which can be used as a typical example of innovative smart (stimuli responsive) materials during college or high school students' education. The preparation of MAC is simple and can be carried out within 2 hours in a common laboratory; the chemicals required are readily available and not expensive. Efficient adsorption of dyes by MAC can be even observed without any photometer, just by visual evaluation; magnetic separation can be performed using cheap, small NdFeB magnets [56].

TABLE IV
APPLICATION OF MAGNETIC ACTIVATED CARBONS (CHARCOAL) FOR THE SEPARATION OF ORGANIC COMPOUNDS

Type of MAC	Separated organic compound	References
Almond shells	2,4,6-Trinitrophenol from water; 97% desorption achieved by methanol and hot water	[44]
Orange peel	Naphthalene and p-nitrotoluene	[45]
Commercial	Methylene blue from river water; maximum adsorption capacity was 47.62 mg g^{-1}	[46]
Hydro-thermal process	Methyl orange from water; maximum adsorption capacity was 44.65 mg g^{-1}	[47]
Coconut shell	Humic substances	[18]
Bitumine	Methylene blue; maximum adsorption capacity was 229.5 mg g^{-1}	[48]
Commercial	Adsorption of methylene blue by activated carbon/cobalt ferrite/alginate composite beads	[31]
Chezacarb B	Water soluble organic dyes from aqueous solutions	[8]
Chezacarb B	Crystal violet and safranin O; magnetic solid-phase extraction used for preconcentration	[49]
Palm shells	Oil from palm oil mill effluent	[50]
Commercial (Norit)	Imidacloprid from water	[51]
Phenolic resin	Methylene orange from water; maximum adsorption capacity was 0.16 mg m^{-2}	[14]
Coconut shell	Methyl orange from water; regeneration by hydrogen peroxide performed	[43]
Rice husk	Methylene blue from water, maximum adsorption capacity was 321 mg g^{-1}	[16]
Commercial	Malachite green from water; maximum adsorption capacity was 89.29 mg g^{-1}	[9]

TABLE V
APPLICATION OF MAGNETIC ACTIVATED CARBONS (CHARCOAL) FOR THE SEPARATION OF INORGANIC COMPOUNDS

Type of MAC	Separated inorganic compound	References
Coconut shell	Mercury; maximum adsorption capacity was 38.3 mg g^{-1}. Hg desorption can be performed by heating	[52]
Bituminous coal	Mercury(II) from water	[53]
Commercial	Arsenic(V) removal from contaminated water with MAC coated with bacteria or biopolymers	[54]
Coconut or fruit pit	Gold from cyanide leach liquor or cyanide pulp	[55]
Orange peel	Phosphate from wastewater	[45]

V. Conclusion

Activated carbons have been used for many applications. Magnetic modifications using various procedures, namely chemical precipitation, high temperature treatment or encapsulation enabled preparation of smart magnetically responsive materials.

This conversion of non-magnetic material into magnetic activated carbon has significantly enriched the fields of practical utilization. Magnetically responsive activated carbons have been successfully used in environmental technology as efficient adsorbents for the removal of wide range of contaminants and pollutants of inorganic (mercury (II), arsenic (V), gold, phosphate, etc.) and organic (water soluble organic dyes, phenolic compouds, humic substances, pharmaceuticals etc.) origin, catalyst supports and materials for magnetic solid-phase extraction. Regeneration enables repeated use of MACs and to keep the operation costs low. In bioapplications, magnetic activated carbons have been used for immunomagnetic assays, immobilization of biologically active compounds or cells and magnetic drug targeting. Definitely, magnetically responsive activated carbons can have many interesting applications in the future.

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