

**ADVANCED MATERIALS OBTAINED BY UNCONVENTIONAL METHODS WITH
APPLICATION INDUSTRIAL AND ENVIRONMENTAL PROTECTION**

Doctoral degree thesis – Summary

Timișoara Polytechnic University

Doctoral field: Chemical Engineering

Ph. D Student: Eng. Istrate Roxana – Georgeta

Scientific coordinator: Prof.univ.dr.eng.Păcurariu Cornelia

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The fast development of some industrial fields and the high priority given to environmental protection have been increasing the interest in obtaining materials with special properties. The field of micro and nanomaterials is at present one of the most dynamic due to the almost unlimited potential applications of these materials.

Industrial use of these materials is conditioned by technical requirements on identifying some synthesis methods to provide the desired properties depending on the application, economical requirements related to production costs and also the capacity of these materials for recycling and re-use.

The purpose of the thesis is the synthesis by unconventional methods of some materials with directed properties for uses in the paint industry and in the environmental protection.

The goals pursued are:

- Synthesis of some luminescent pigments used in order to obtain luminescent paints for industrial and security markings, and respectively
- Synthesis of some advanced adsorbent materials, in order to reduce the costs of wastewater treatment by increasing the adsorption capacity.

Thus, a luminescent pigment based on strontium aluminate doped with Eu and Dy was synthesized, used in order to obtain luminescent paints for marking in educational, leisure or industrial areas.

This pigment was obtained by combustion method, a method which has not been mentioned in literature for this purpose, which is easy, environmentally friendly and allows a substantial decrease in the cost price as a result of the obtaining of the compound directly by synthesis reaction, without the need for further thermal treatments.

New adsorbent materials have also been synthesized which are cheap, renewable and reusable, with wide-ranging capability, for the removal of some pollutants from the

wastewater. For this purpose there were obtained, by solvothermal method and also by combustion, nanocomposites with magnetic properties based on iron oxides and carbon that have been tested as adsorbents to remove some dyes, phenol and some phenol derivatives, from water. A polymeric adsorbent has also been synthesized and tested in order to remove different pollutants from wastewater.

CHAPTER 1 LUMINESCENT PIGMENTS

Increasing safety in various areas such as storage of hazardous products, public spaces (kindergartens, schools, hospitals, airports, shopping centers, gyms), industrial halls and road traffic is a major objective. The minimum requirements for safety and / or health signaling at the workplace are regulated by GD 971/2006 (updated on 1 June 2015), which transposes Directive 92/58 / EEC published in the Official Journal of the European Communities (JOCE) no. L 245/1992. As a result of these requirements, the increase in interest in luminescent paints is fully justified.

The enormous potential of luminescent paints is proven by their use for markings in the dark of the so-called "highways of the future". The bright markings use luminescent paints that are charged with solar energy during the day and then light up to 10 hours when it is dark. These give more visibility and safety to roads than those marked with standard paints and, moreover, do not involve electricity consumption. Such a highway was opened in 2014 in the Netherlands.

The particularly high potential of luminescent paint applications explains the increasing interest in obtaining luminescent pigments by using environmentally friendly, simple, cost-effective synthesis methods that maximize their luminescent properties [1].

CHAPTER 2 ADVANCED MATERIALS USED AS ADSORBENTS FOR THE REMOVAL OF SOME POLLUTANTS FROM WASTEWATER

Population growth and rapid and continuous industrialization globally have a negative impact on the quality of the environment. Water is one of the most important elements that sustain life [2]. Numerous industries such as mining, textiles, nuclear, petroleum, dyes and paints, paper, leather and others are large generators of polluted effluents with devastating effects on human health and the environment [3]. For this reason, governmental legislation is ever stricter on the rational use of water sources and the finding of the most appropriate methods for removing pollutants from waste water in order to ensure its superior quality. Therefore, the development of economical and efficient wastewater treatment methods to bring the concentration of pollutants within the limits imposed by environmental regulations is a constant concern.

In the last decades, a wide variety of contaminated water treatment techniques have been developed [4, 5]. The most important of these are: solvent extraction [6], micro and ultrafiltration, sedimentation and gravity separation, floating, precipitation, coagulation, oxidation, reverse osmosis, adsorption, ion exchange, electro dialysis, electrolysis, photocatalytic degradation, bioremediation, etc. [7,8]. These methods differ from one another in terms of cost, efficiency and environmental impact. The chosen method has to be as efficient as possible to remove a pollutant from contaminated water and be available at the lowest cost [9]. From this point of view, adsorption is cheaper than other water treatment methods [10, 11].

Besides low cost, adsorption is one of the most important and competitive techniques for wastewater treatment due to its easy operation, high efficiency, variety of available adsorbents, and the possibility of regeneration and reuse in multiple adsorption-desorption cycles, with the possibility of recovering the pollutant if it is of interest. On the other hand, adsorption can be applied to eliminate a wide range of soluble or less soluble, organic, inorganic or biological pollutants [12, 13].

CHAPTER 3 SYNTHESIS OF SOME LUMINESCENT PIGMENTS BY THE COMBUSTION METHOD AND THEIR TESTING IN PAINTS

In this chapter it was studied the production of SrAl_2O_4 by the combustion method using two different approaches: the classic one by using a single fuel (urea or glycine) and a new, original approach based on the use of a mixture of fuels (urea and glycine).

It has been demonstrated that the classic single-fuel approach (urea or glycine) does not result in the production of SrAl_2O_4 directly from the combustion process. Thermal images have shown that the maximum measured temperature is much lower than the calculated adiabatic temperature, being insufficient to ensure SrAl_2O_4 formation, as also shown by the RX diffraction analysis and FTIR spectrometry.

The original approach to the blend of fuels (urea and glycine) had a remarkable effect on the maximum measured temperature that is much closer to the calculated adiabatic temperature and high enough to ensure direct formation of SrAl_2O_4 without subsequent thermal treatments.

Samples obtained using the fuel mixture contain a mixture of $\alpha\text{-SrAl}_2\text{O}_4$ as the main phase and $\beta\text{-SrAl}_2\text{O}_4$ as the secondary phase.

It has been shown that adding a small amount of H_3BO_3 to the precursor solution results in the formation of $\alpha\text{-SrAl}_2\text{O}_4$ as a single phase directly from the combustion reaction without further thermal treatments.

The obtained results demonstrate the major importance of proper fuel selection by taking into account the ability of the metallic-fuel nitrate systems to generate the critical fuel gas concentration, which subsequently leads to ignition and thus to the formation of the desired compounds.

A luminescent pigment based on SrAl_2O_4 doped with Eu and Dy was obtained by the combustion method using a fuel mixture (urea and glycine) without additional thermal treatments. To achieve a reducing atmosphere an excess of urea was used. The excitation spectrum shows a maximum at 331 nm and a emission at 518.5 nm, confirming the green color of the pigment emission obtained.

The emission time of the pigment is 1800 s, while the visible emission is approximately 12 minutes.

The obtained pigment retains its luminescent properties after the UV light has ceased.

The pigment obtained by the combustion method has been successfully used to obtain paints with luminescent properties in order to be used for industrial markings and warning signals, demonstrating the possibility of using it in industrial applications.

The used combustion method has significant advantages compared to the results presented in the literature; in addition to the low temperatures required for synthesis, the short time required to obtain SrAl_2O_4 leads to a considerable decrease in operational costs. Thus, the temperatures required to obtain SrAl_2O_4 reported in the literature when using the solid state reaction range between 900-1300°C [14-16], significantly higher compared to combustion synthesis (using the fuel mixture) used in these investigations (450°C). Also, using the co-precipitation method proposed by Bandy et al. [17], the temperature required to obtain SrAl_2O_4 was 800 ° C, and the required time was 12 hours (much longer than the combustion method used in the thesis - 5 min).

CHAPTER 4 SYNTHESIS, CHARACTERIZATION AND TESTING OF SOME IRON OXIDE/ CARBON NANOCOMPOSITES AS ADSORBANTS FOR THE REMOVAL OF SOME POLLUTANTS FROM WATER

Obtaining of adsorbing materials with high adsorption capacity, low cost, easy separation of the phases, regeneration and reuse capacity in multiple adsorption-desorption cycles and possibilities for industrial scale use shows particular interest and a permanent concern. In this context, the magnetic nanocomposites iron oxide/carbon ensure both easy separation of the phases by means of a magnet and a high adsorption capacity due to the presence of carbon.

In this chapter are presented the results obtained in the synthesis of magnetic

nanocomposites using two different nonconventional methods: Solvothermal method, using different precipitation agents (KOH or diethylamine) as well as combustion method. The obtained magnetic powders were tested in the adsorption process of a single pollutant (phenol, some derivatives of phenol, methyl orange (anionic dyes), methyl blue (cationic dye)) as well as simultaneous adsorption in a binary system (phenol and methyl Orange).

A study has been carried out to obtain magnetic nanocomposites, iron oxide/carbon, using different synthesis methods (solvothermal and combustion) and various synthetic conditions.

The nanocomposites obtained have been characterized and tested as adsorbent materials for a wide range of pollutants, namely: anionic dyes (MO), cationic dyes (MB), phenol and phenol derivatives such as p-chlorophenol, 3-aminophenol, p- Dimethyl-phenol and trimethyl-phenol.

A single pollutant adsorption study (MO or phenol) and simultaneous adsorption of both pollutants (MO and phenol) were conducted.

Iron oxide/carbon nanocomposites with a molar ratio ranging from 1/1 to 1/2 were prepared by the solvothermal method using KOH as precipitant and 1,2-propanediol as a solvent.

The doubling of the carbon content from the MNP2 sample to the MNP3 sample determined the area of the specimen area to be increased from 189.4 m² g⁻¹ to 394.06 m² g⁻¹.

It has been shown that the efficiency of the removal of MO and phenol can be significantly improved by increasing the active carbon content of magnetic nanopowders which can be correlated with the much higher specific surface area of the MNP3 adsorbent compared to MNP2. Thus, for an adsorbent mass of 2 g L⁻¹, the MO removal efficiency was 99% for the MNP3 adsorbent and 67% for the MNP2 adsorbent.

It has been demonstrated that the adsorption of MO and phenol is described by pseudo-second order kinetics, both with the use of a single pollutant (MO or phenol) and with the use of a mixture of pollutants (MO and phenol).

In the case of adsorption of a single pollutant, it was demonstrated that both adsorbents had an adsorption capacity of 1.7 times higher in the case of MO than in the case of phenol, which can be explained by the much lower MO solubility (5 g L⁻¹) in water as compared to phenol (83 g L⁻¹).

Of the four investigated isotherms, Langmuir, Freundlich, Redlich-Peterson and Sips, in the case of simple adsorption, the equilibrium data are best correlated with the Langmuir isotherm for both MO and phenol on MNP2 and MNP3 adsorbents. The maximum adsorption

capacity in the monomolecular layer was 72.68 mg L^{-1} and 42.34 mg L^{-1} , respectively, for the addition of MO and phenol respectively on the MNP3 adsorbent. The simultaneous adsorption process in the MO-Ph binary system is best described by the Langmuir isotherm, and in the Ph-MO binary system is described by the Sips isotherm.

Regarding the simultaneous adsorption of the two pollutants, it was established that the adsorption of MO is not substantially affected by the presence of phenol, whereas the phenol adsorption capacity is significantly reduced by the presence of MO. This behavior was explained on the basis of the much stronger attraction between MO molecules and the surface of the adsorbent, involving both electrostatic and non-electrostatic interactions, whereas the phenol adsorption mechanism does not involve electrostatic interactions.

Desorption studies carried out both on the use of a single pollutant (MO or phenol) and on the use of a mixture of pollutants (MO and phenol) have shown a good capacity for regeneration and reuse of the MNP3 adsorbent.

It has been shown that using diethylamine instead of KOH as precipitating agent in the solvothermal method, magnetic nanocomposites iron oxide/carbon have been obtained whose textural characteristics and adsorbent properties are not substantially altered.

The FTIR analysis revealed the presence of magnetite as the main phase along with maghemite in all samples, this being in a larger proportion in the case of coal nanocomposites. The Mössbauer spectra have highlighted the complete transition, in time, of magnetite into magemite.

The synthesized nanocomposites were investigated as adsorbent materials for the elimination of a cationic dye (methyl blue) from aqueous solutions, demonstrating the improvement in removal efficiency with the increase in the carbon content of the samples.

Kinetic studies have shown that the adsorption process of methyl blue is described by pseudo-second-order, and the equilibrium data correlated with the Reddlich-Peterson isotherm.

Adsorption studies have demonstrated an increase in the maximum MB adsorption capacity due to the increase in the carbon content of nanocomposites; thus, the maximum blue methyl adsorption capacity, determined on the basis of the Langmuir isotherm, increased from 34.81 mg g^{-1} in the $\text{Fe}_x\text{O}_y/\text{C}$ adsorbent to 60.84 mg g^{-1} in the case of $\text{Fe}_x\text{O}_y/2\text{C}$ adsorbent.

The effectiveness of using magnetite / activated carbon nanocomposites synthesized by the combustion method to eliminate phenol and its derivatives in aqueous solutions has been demonstrated.

Increasing the carbon content from 1: 3 for M1-C3 to 1:10 for M1-C10 led to an increase in elimination efficiency for both phenol and its derivatives. This behavior can be explained by increasing the surface area specific for the adsorbent M1-C10.

It has been demonstrated that both adsorbents, M1-C3 and M1-C10, with different charcoal content, have a high adsorption capacity for less soluble pollutants and lower pK_a values.

Increasing the final concentration of pollutants has increased the amount of adsorption at equilibrium; less for phenol and more for p-NP.

The adsorption process is very fast; the contact time required to achieve the equilibrium was approximately 30 min for p-NP and 240 min for phenol and 3-AP when using M1-C3 as the adsorbent.

The kinetics of the adsorption process of phenol, 3-AP and p-NP on the M1-C3 adsorbent is described by pseudo-second order. It has been established that the rate of adsorption process increases in the order: phenol<3-AP<p-NP.

The phenol adsorption process is best described by the Langmuir isotherm, 3-AP adsorption of the Sips isotherm and the p-NP adsorption of the Redlich-Peterson isotherm. By comparing the maximum adsorption capacities obtained from the Langmuir isotherm and the Sips isotherm for the three pollutants, the adsorption capacity of adsorbent M1-C3 is confirmed in the order: phenol<3-AP<p-NP.

It has been demonstrated that both the solvothermal method and the combustion method are simple, cheap and efficient methods of obtaining iron / carbon oxide nanocomposites with remarkable adsorption capacity to eliminate a wide range of pollutants from aqueous solutions.

The unique combination of high adsorption capacity, excellent separation capacity, short time needed to achieve balance, and low cost cost are features that indicate that iron / carbon oxide magnetic nanoparticles studied are excellent adsorbents with potential for wastewater treatment on an industrial scale.

CHAPTER 5 SYNTHESIS, CHARACTERIZATION AND TESTING OF SOME COPOLYMERS AS ADSORBENTS FOR THE REMOVAL OF SOME POLLUTANTS FROM WASTEWATER

Polymeric adsorbents, including polystyrene-divinylbenzene copolymers, are considered chemically inert, stable, highly adsorption-efficient, selective, with relatively low costs and limited toxicity [18-20]. The adsorption capacity of polymeric adsorbents for a specific

pollutant can be improved by introducing special functional groups into the adsorbent matrix [20].

In this chapter the synthesis, characterization and testing of a styrene-divinylbenzene copolymer functionalized with carboxyl groups as an adsorbent for the elimination of some pollutants from aqueous solutions was followed. Testing of the obtained copolymer as an adsorbent to remove p-nitrophenol (p-NP), methyl orange (MO) and 3-aminophenol (3-AP) from aqueous solutions [19, 20] was also followed.

The feasibility of the surface function of the styrene-divinylbenzene chloromethylated copolymer has been demonstrated to improve the adsorption of p-NP, MO and 3-AP in aqueous solutions. The presence of carboxyl groups on the surface of the CP-F copolymer as a result of functionalization results in a significant increase in adsorption capacity compared to the non-functional polymer.

The mechanism of p-NP adsorption on the non-functionalized (CP-N) and functionalized carboxyl groups (CP-F) was discussed, making it clear that the easy formation of hydrogen bonds is the main explanation.

It has been demonstrated that increasing the functionalized copolymer mass, CP-F, from 1 g L⁻¹ to 5 g L⁻¹, increases the efficiency of p-NP removal from 56% to 92%.

It was established that p-NP adsorption kinetics on CP-F are described by pseudo- second order model and equilibrium data correlated with the Langmuir, Freundlich, Redlich-Peterson and Sips isotherms in the following order: Sips > Redlich-Peterson > Langmuir > Freundlich.

The maximum adsorption capacity of the CP-F polymer resulting from the Sips isotherm was 243.34 mg g⁻¹, which is higher compared to other literature reported on the adsorption of p-NP on different adsorbents.

It has been demonstrated that the styrene-divinylbenzene copolymer functionalized with carboxyl groups has good adsorption capacity for other pollutants such as anionic colorant MO and 3-aminophenol.

Kinetic studies have indicated that both MO and 3-AP adsorption on the functionalized copolymer follows the pseudo-second order model.

It was determined that the Redlich-Peterson isotherm best describes the adsorption of MO on the CP-F copolymer while the 3-AP adsorption is described by the Freundlich isotherm.

Comparing the maximum adsorption capacities obtained from the Langmuir isotherm for the three pollutants p-NP (219.45 mg g⁻¹), MO (53.12 mg g⁻¹) and 3-AP (47.19 mg g⁻¹) it can be seen that the best adsorption capacity of the CP-F polymer was obtained in the case of p-NP elimination.

The regeneration and re-use studies of the CP-F polymer have determined that the efficiency of p-nitrophenol elimination after three adsorption-desorption cycles increases with the increase in the amount of adsorbent while the desorption time period has virtually no influence.

The results from this study demonstrate the possibility of successfully using the styrene-divinylbenzene chloromethylated copolymer, functionalized with carboxyl groups, as an adsorbent for the removal of p-NP, MO and 3-AP from aqueous solutions, but also the good ability to regenerate and reuse suggesting its potential use on a large scale.

CHAPTER 6 GENERAL CONCLUSIONS

Strontium aluminate was synthesized for the first time by the combustion method using a mixture of fuels (urea and glycine) without additional thermal treatments.

The importance of choosing the right fuel has been demonstrated by taking into account the ability of metallic fuel-to-fuel systems to generate critical fuel gas potential, which subsequently leads to combustion and the formation of the desired compounds.

A luminescent pigment based on strontium aluminate doped with Eu and Dy was obtained by combustion method using a mixture of fuels (urea and glycine).

The pigment has been successfully tested in order to obtain paints with luminescent properties for industrial markings and signage marks.

Iron oxide/carbon nanocomposites were synthesized by solvothermal method using KOH as a precipitating agent without additional thermal treatments.

The iron oxide/carbon nanocomposites obtained by the solvothermal method using KOH precipitating agent have been tested as adsorbent materials for the removal of MO and phenol from aqueous solutions in both simple and simultaneous adsorption.

It has been shown that by increasing the amount of active carbon, the adsorption capacity of nanocomposites can be considerably improved.

Iron oxide/carbon nanocomposites were synthesized by solvothermal method using diethylamine as precipitating agent without further treatment.

The iron oxide / charcoal nanocomposites synthesized by the solvothermal method, using diethylamine as the dopant, have been successfully used to remove MB from aqueous solution.

It has been pointed out that the adsorbent properties and textural characteristics of the iron oxide / carbon monoxide nanocomposites are virtually unchanged by the use of diethylamine instead of KOH as a precipitating agent in the solvothermal method.

Increased adsorption capacity has been shown to increase the amount of activated carbon to remove phenol and phenol derivatives using iron oxide/carbon adsorbents with different carbon content, obtained by the combustion method.

A high adsorption capacity of iron oxide/carbon nanocomposites has been demonstrated for less soluble pollutants and lower pK_a values.

The capacity of regeneration and reuse of iron oxide/carbon nanocomposites in multiple adsorption-desorption cycles was highlighted.

It has been demonstrated the importance of functionalizing the surface of the styrene-divinylbenzene chloromethylated copolymer to improve the adsorption of p-NP, MO and 3-AP in aqueous solutions.

It has been demonstrated the possibility of increasing the adsorption capacity of polymers by directed functionalization of the surface due to strong interactions between the adsorbent and a specific pollutant.

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