Removal of two ionic dyes from water by MgO-loaded porous carbons prepared through one-step process from poly(ethylene terephthalate)/magnesium carbonate mixtures

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Abstract. Mixtures of poly(ethylene terephthalate) and magnesium carbonate at different weight ratios were heated up to 850°C in argon atmosphere. During heating, components of the initial mixtures underwent thermal decomposition yielding porous carbon materials loaded with different amounts of magnesium oxide. Structural characteristics of the prepared materials were determined from adsorption/desorption isotherms of nitrogen, measured at 77 K. For reference, portions of the products obtained were acid-washed to obtain MgO-free carbons. Pore structures of the prepared materials were strongly dependent on the quantitative compositions of starting mixtures. As a rule, specific surface areas determined for acid-washed materials were much higher than those for MgO-loaded carbons. The adsorption abilities of obtained materials towards cationic (Basic Red 18) and anionic (Reactive Red 198) dyes as model contaminants were examined. Surprisingly, in spite of relatively low specific surface areas, substantially high adsorption of the dyes on MgO-loaded carbons was observed. An influence of specific surface area and the role of magnesium oxide presence on the adsorption capacity of the acquired sorbents were studied.

Keywords. Carbon material; magnesium oxide; PET; adsorption; reactive red 198; basic red 18.

1. Introduction

Activated carbon is one of the most frequently employed adsorbent in many applications (Przepiórski 2006). This material is produced mainly from natural precursors like wood (Acharya *et al* 2009; Girods *et al* 2009; Yorgun *et al* 2009) or coals (Girods *et al* 1996; Lázaro *et al* 2007; Lillo-Ródenas *et al* 2007; Zhang *et al* 2008). Nevertheless, there are many studies on the preparation of carbonaceous adsorbents from pitches (Przepiórski *et al* 1999) or synthetic materials including phenolic resins (Cai *et al* 2004; Nakagawa *et al* 2007; Gun'ko *et al* 2008; Kubota *et al* 2009), polyacrylonitrile (László *et al* 1999, 2000, 2001; Zaini *et al* 2010) or polyethylene terephthalate (László *et al* 1999, 2000, 2001; Parra *et al* 2004; Kartel *et al* 2006; Almazán-Almazán *et al* 2010). Utilization of these materials as carbon precursors is often related to the alternative methods for disposal of plastic wastes.

Normally, preparation of porous carbons from polymeric precursors involves conventional carbonization and subsequent activation (László *et al* 1999, 2000, 2001; Parra *et al* 2004; Kartel *et al* 2006; Almazán-Almazán *et al* 2010). However, numerous works deal with one-step process, usually consisting in the thermal treatment of carbon precursors with specific additives, playing a role of pore creating agents

(Inagaki *et al* 1998, 1999, 2004; Konno *et al* 2000; Nakagawa *et al* 2003; Terakado and Hirasawa 2005; Morishita *et al* 2006; Przepiórski *et al* 2009). Thus, in addition to a char formed from the carbon precursors, the purposely used additives may become an important component of the final product. In this way, a range of additive-loaded porous carbons for various applications can be obtained (Przepiórski 2006).

An interesting method of obtaining porous carbons from polymers without any activation process was reported by Inagaki *et al* (1998, 1999, 2004), Konno *et al* (2000), Morishita *et al* (2006). The authors proposed pyrolysis of various ceramic materials such as certain metal oxides or other species, coated with carbon precursors. In all the studies, the inorganics remained in the product after the thermal treatment were dissolved through washing with acid. The carbon materials prepared in this manner revealed well-developed porosity with a significant contribution of mesopores together with micropores. Applying related procedures, it was possible to obtain nanoporous carbons by using magnesium oxide (Inagaki *et al* 2004; Przepiórski *et al* 2009) or some thermally decomposable magnesium compounds (magnesium acetate, magnesium citrate, magnesium gluconate and magnesium carbonate), considered as MgO precursors (Morishita *et al* 2006; Inagaki *et al* 2007; Przepiórski *et al* 2009). Because of bimodal, i.e. microporous and mesoporous character, an ability of such porous carbons to remove pollutants of various molecular sizes may be expected. It

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should be noticed that magnesium oxide itself is capable to remove certain contaminants from water. As reported elsewhere (Nagappa and Chandrappa 2007; Moussavi and Mahmoudi 2009), MgO efficiently eliminated fluoride ions as well as anthraquinone and azo dyes. For instance, adsorption of the Reactive Red 198 on MgO nanoparticles was found to be comparable to that on some activated carbons.

From the information given above, one may conclude that a hybrid material combining features of both porous carbon and MgO might also be capable of eliminating dyes from water efficiently. Hence we have prepared porous carbon loaded with MgO in one-step process. The product obtained was examined as a potential sorbent for removal of both anionic (Reactive Red 198) and cationic (Basic Red 18) dyes from water.

2. Experimental

2.1 Materials

Poly(ethylene terephthalate) (PET) used for sorbent preparations was of commercial grade, obtained from Elana Ltd. (Poland). Another raw material, MgCO_3 , of reagent grade, was purchased from Sigma-Aldrich Co. (Germany). A commercially available activated carbon CWZ-35 produced according to a conventional process using steam as an acti-

vating medium, was used as a reference material. This activated carbon was obtained from Gryfskand Ltd. (Poland). High purity (99.999%) argon gas purchased from Messer (Austria) was used during preparations. Commercial grade anionic (Reactive Red 198) and cationic (Basic Red 18) dyes, were used as model adsorbates. The dyes were obtained from Boruta-Kolor Ltd. (Poland). The characteristic data and chemical structures of these dyes are shown in table 1 and figure 1, respectively.

2.2 Preparations

Carbonaceous sorbents containing magnesium oxide were prepared through a pyrolysis of homogenized mixture of precursor materials. At first, powdered portions of magnesium carbonate (MgCO_3 , magnesium oxide precursor) and PET (carbon precursor) at three weight ratios: 30/70, 50/50, and 70/30, were mechanically agitated. In order to achieve an effective homogenization, obtained MgCO_3 /PET mixtures (5 g per lot) were placed in a tubular furnace and first round heated under argon flow at 265°C (1 h), which resulted in melting of PET contained in the mixtures. After cooling, obtained solid products were ground and then again subjected to the heating/grinding procedure as described above. Finely ground mixtures obtained in this method served as raw materials to prepare sorbent materials. Consequently, 5 g of each raw powder was placed in a tubular furnace and heated up to 850°C ($10^\circ\text{C}/\text{min}$) under argon gas flow ($25\text{ cm}^3/\text{min}$) and the final temperature was maintained for 1 h. Then, the system was allowed to cool down which took *ca.* 2 h. The products attained were pulverized and finally stored in a tight container. For comparison purposes certain amounts of the prepared materials were additionally acid-treated to wash out inorganics contained in the product and thus MgO-free carbon material could be obtained. For that purpose, an excessive amount (*ca.* 100 cm^3) of 3 M HCl solution was used at first, and then the carbonaceous products were rinsed with distilled water until constant pH was achieved. Thus, two series of samples were obtained i.e. non-acid washed and acid washed, labelled in this work as NAW and AW, respectively. Furthermore, portions of sole MgCO_3 and PET were separately subjected to heat treatment in the similar manner that was used for the treatments of raw MgCO_3 /PET mixtures. Thus, two additional materials were obtained i.e. MgO (product of MgCO_3 thermal decomposition) and a char (product of PET pyrolysis).

2.3 Methods

The X-ray diffraction patterns were measured using Philips X'Pert PRO diffractometer operating with the CuK_α radiation. The thermogravimetric (TG) analyses were carried out on STA 449 C TGA apparatus (Netzsch, Germany). Measurements were performed in argon atmosphere ($25\text{ cm}^3/\text{min}$) in two steps. At first, samples were dynamically heated from room temperature up to 850°C with heating rate $10^\circ\text{C}/\text{min}$. Then followed by an isothermal step,

Figure 1. Chemical structures of used dyes.

Table 1. The characteristics of used dyes.

Dye	Abbreviation	CAS number	C.I. number	Molecular weight	$\lambda_{\max}$ (nm)
Reactive Red 198	RR198	145017-98-7	18221	968.21	516
Basic Red 18	BR18	14097-03-1	11085	390.89	480

where the final temperature (850°C) was maintained for 1 h. In order to determine MgO contents in the obtained products, additional TG measurements were necessary to be carried out in air atmosphere (25 cm³/min) from 25°C to 850°C (heating rate 10°C/min). This procedure allowed the burning out of carbon material formed from PET during heating under argon gas, and thus contained the obtained hybrid product together with MgO. Hence, the residual masses after TG measurements were assumed to be directly related to the MgO contents in the prepared materials.

The nitrogen adsorption/desorption isotherms at 77 K were determined with use of Quadrasorb SI (Quantachrome Instruments, USA). Prior to analyses, each sample was degassed at 290°C for 24 h under high vacuum. The values of the specific surface areas (S_{BET}) were determined using multi-point analysis of adsorption isotherms applying Brunauer-Emmet-Teller (BET) equation. In addition, micropore surface areas (S_{micro}), external surface areas (S_{ext}) and total surface areas (S_{total}) were estimated using α_s method.

The adsorption capacities of obtained materials toward RR198 and BR18 were determined by applying a batch method. Specific amounts (10–50 mg) of tested material were added to 100 cm³ of aqueous solution (30 mg/dm³) of each dye and obtained suspensions were thermostated in a shaking bath at 30°C. After 2 h, concentrations of the dye in each system were determined spectrophotometrically by using UV-Vis spectrophotometer (V-530, Jasco, Japan).

In addition, the values of zeta potential for each material was measured using Zetasizer Nano Series apparatus equipped with an autotitrator (Malvern Instruments, UK). The zeta potential measurements were performed within pH range of 5–10.5. The pH was automatically adjusted by the addition of either 0.025 M HCl or 0.05 M NaOH solutions.

Because adsorption tests were carried out in a water environment, some of MgO contained in the prepared hybrid materials might be lost due to the dissolution. Hence, in order to estimate an extent of MgO elution, Mg contents in a water phase were analysed after adsorption tests. For that purpose, an inductively coupled plasma optical emission spectrometer (Optima 5300DV ICP-OES, Perkin Elmer, USA) was used and amounts of MgO eluted could be easily calculated.

3. Results and discussion

3.1 XRD analysis

An example of the X-ray diffraction pattern measured for NAW-series products is presented in figure 2.

Six well-defined peaks, confirming the presence of MgO could be observed on the pattern. Similar results (not shown here) were obtained for other samples from the series. Presence of MgO in the structures of these samples is quite expectable and stays in accordance with the results of others (Khan *et al* 2001), confirming thermal decomposition of MgCO₃ to the oxide at *ca.* 550°C.

3.2 Carbon and magnesium oxide contents in NAW-series materials

Amounts of carbonaceous material and magnesium oxide determined for as-prepared NAW-series samples are compiled in table 2. As evident, contents of MgO in the obtained products tend to increase along with MgCO₃ loading in the initial mixtures, and reaches *ca.* 46 wt.% at the maximum. Obviously, as NAW-series samples consist of MgO and carbon material, the increase in MgO content is connected with a complementary decrease in carbon contents.

As the materials were intended to be tested as adsorbents in a water environment, amounts of magnesium oxide possibly eluted from the samples due to hydrolysis or dissolution had to be evaluated. Concentrations of magnesium in a water phase, after separation of solid, were close to the expected values as resulting from the solubility of MgO in water (86 mg/dm³ at 30°C, Patnaik 2003). This had an impact on the MgO loading in NAW-series samples subjected to adsorption test. As shown in table 2, adsorption of dyes from water is burden with 7–8 wt. % loss due to MgO elution. Taking into account the total MgO loading, this loss can be

Figure 2. XRD pattern of NAW carbon obtained from MgCO₃/PET (70/30) mixture after heating at 850°C.

Table 2. Contents of carbon material and MgO in the prepared materials before and after adsorption tests (in wt. %).

MgCO ₃ /PET weight ratio in raw mixture	Carbon content	MgO content before adsorption test	MgO loss during adsorption test	MgO content after adsorption test
30/70	78.73	21.27	7.20	15.16
50/50	69.09	30.91	8.40	24.57
70/30	53.75	46.25	7.10	42.14

considered as substantial in the case of samples less loaded with MgO or less important for materials richer in MgO.

3.3 Nitrogen adsorption/desorption (77 K) isotherms analysis

Nitrogen adsorption/desorption isotherms measured at 77 K, for NAW- and AW-series samples as well as for commer-

cially available CWZ-35 activated carbon are presented in figure 3.

The shapes of all the isotherms measured for the prepared samples, at a low relative pressure region, are of type I (according to IUPAC classification) and indicate the presence of micropores. In addition, all the isotherms reveal distinctive hysteresis, like in type IV isotherms, between adsorption and desorption branches at relative pressures p/p_0 above 0.5 (NAW-series samples) and 0.4 (AW-series samples), indicating capillary condensation in mesopores. Hence, the shapes

Figure 3. Nitrogen adsorption/desorption isotherms (measured at 77 K) on: (a) NAW materials, (b) AW materials and (c) commercial activated carbon (CWZ-35).

Table 3. Pore structure parameters of obtained materials and commercial activated carbon, determined using BET and α_s plot methods.

	MgCO ₃ /PET ratio in starting mixture	α_s analysis			
		S_{BET} (m ² /g)	S_{total} (m ² /g)	S_{ext} (m ² /g)	S_{micro} (m ² /g)
NAW carbons	30/70	383	477	204	273
	50/50	284	349	157	192
	70/30	141	156	106	50
AW carbons	30/70	835	946	562	384
	50/50	1120	1196	843	353
	70/30	1781	1829	1581	248
MgO	100/0	49	–	–	–
Char	0/100	239	–	–	–
CWZ-35 (commercial activated carbon)	–	1260	1494	335	1159

of the isotherms formed are due to the overlapping of type I and IV isotherms and thus confirming large share of micropores and mesopores in the total porosity of the samples. Large contribution of both classes of pores may be explained by the action of MgO and CO₂ (products of MgCO₃ decomposition) on char formed from PET, as it was explained in detail in our earlier work (Przepiórski *et al* 2009). The isotherms collected for the CWZ-35 activated carbon indicate its predominant microporous character with less mesoporosity. Different courses of the measured isotherms are reflected by appropriate differences in pore parameters (table 3).

In general, compared to acid-washed materials (AW), NAW-series materials reveal rather low N₂ uptake (figure 3). Hence, all the pore parameters calculated for NAW-series materials are much lower than the others. This particularly concerns mesopore area and to a lesser extent micropore area. From the pore structure parameters and MgO loadings (table 3) within NAW-series materials, it is evident that

mesopore and micropore areas tend to decrease on increasing MgO contents. On the other hand, washing out MgO, results in materials with pore structure parameters that tend to increase with MgO loadings in initial samples (before acid treatment). This indicates a strong impact of MgO presence in samples on nitrogen uptake and therefore on pore parameters. Such tendency must be resulting from a well-known pore-blocking effect caused by MgO deposits. Apparently, washing out MgO from NAW-series samples results in new products, free of pore-blocking effect, and thus with pores opened to the N₂ molecules. Because of this, adsorption of nitrogen on MgO-free samples is generally much higher than on those loaded with the oxide. Similar phenomena occurring in a range of various additive-loaded porous carbons were reported in numerous works (Inagaki *et al* 1999; Maroto-Valer *et al* 2005; Carrott *et al* 2006).

From the data presented above, it may be stated that nitrogen uptake by all the individual samples also depends on MgCO₃/PET ratio in the starting materials. However,

Figure 4. Removal of Reactive Red 198 from water in the presence of: (a) NAW materials, MgO or char and (b) AW materials or commercial activated carbon (CWZ-35).

while pore parameters tend to decrease with the ratio for NAW-series material, porosity of AW-series samples greatly increase with the ratio.

3.4 Adsorption ability of the obtained carbons

To determine that the MgO-loaded porous carbons (NAW) show a potential to purify water, adsorption of two model adsorbates (RR198 and BR18) on the materials, was carried out. In addition, performances of pure MgO and char, prepared by a thermal decomposition of MgCO_3 and pyrolysis of PET, respectively, were also examined. Adsorption of the dyes on AW-series carbons and CWZ-35 commercial activated carbon were compared and the results are presented in figures 4 and 5.

In general, NAW-series sorbent materials in spite of rather low surface area (table 3) are capable of removing both dyes from water. However, while extent of RR198 adsorption substantially increases with MgO loading in a sorbent, this factor has only slight influence on the removal of BR18. Surprisingly, MgO itself revealing very low specific surface area, but efficiently sorbs RR198, while removal of BR18 by the oxide is negligible. On the other hand, the char produced through PET pyrolysis cannot be considered as an efficient sorbent for any of the tested dyes.

As seen from table 3 and figures 4 and 5, washing out MgO, results in new highly porous sorbents showing somewhat decreased performance during RR198 removal but showing highly increased adsorption of BR18. From the above observations, it was difficult to specify which is the key factor to influence the removal of the dyes by sorbents loaded with MgO. Since RR198 is an anionic dye and BR18 is a cationic one, influence of another factor possibly favouring sorption on NAW materials was considered. One should notice that the adsorption in general is influenced by repulsive and attractive forces, resulting from an

Table 4. Autogenous pH of respective sorbent/dye combinations.

Sorbent/dye combination	Autogenous pH
AW (30/70)/RR198	6.3
AW (50/50)/RR198	6.5
AW (70/30)/RR198	6.6
NAW (30/70)/RR198	9.9
NAW (50/50)/RR198	9.8
NAW (70/30)/RR198	10.2
MgO/RR 198	10.4
AW (30/70)/BR18	5.4
AW (50/50)/BR18	5.1
AW (70/30)/BR18	6.0
NAW (30/70)/BR18	9.7
NAW (50/50)/BR18	9.3
NAW (70/30)/BR18	9.6
MgO/BR18	10.2

interaction of charges between an adsorbent and an adsorbate. This also concerns our adsorption systems, employing both NAW- and AW-series materials, suspended in aqueous solutions of the dyes. A convenient parameter to know the susceptibility of a surface to become either positively or negatively charged, is the zeta potential value. Since the zeta potential is a pH-dependent parameter, it was necessary to determine adsorbent–adsorbate relation in pH range that were achieved during adsorption tests. As shown in table 4, pH values measured for each sorbent/dye combination after adsorption test, ranged from *ca.* 5 to *ca.* 10.5, and were higher for systems employing sorbents loaded with MgO. Moreover, when magnesium oxide alone was used, pH was still higher and reached *ca.* 10.2 and *ca.* 10.4 for BR18 and RR198, respectively. This tendency is quite expectable, and must be related to a weak alkalization of the system as a

Figure 5. Removal of Basic Red 18 from water in the presence of: (a) NAW materials, MgO or char and (b) AW materials or commercial activated carbon (CWZ-35).

Figure 6. Zeta potential vs pH curves of used materials: (a) AW materials, (b) NAW materials and (c) dyes.

result of MgO hydrolysis. For these reasons, the zeta potential values were measured at pH ranging from *ca.* 5 to *ca.* 10.5 and the results are presented in figure 6.

To estimate if adsorbent–adsorbate combinations employed by us are favorable for adsorption or not, differences in the potentials measured for components of each sorbent/dye combination were calculated using data presented in figure 6 and the results are plotted in figure 7.

Removal of the dyes by the pure MgO with a very low specific surface area, can be considered as a good illustration how the electrostatic forces influence on adsorption. Hence, quite efficient elimination of RR198 from water can be attributed to the relatively big difference in zeta potentials between MgO and the dye (60.5 mV at autogenous pH, *ca.* 10.4). On the other hand, much smaller difference in the potentials measured for MgO and BR18 (*ca.* 18.1 mV, at autogenous pH, *ca.* 10.2) is reflected by a minor effect of the oxide on the concentration of this dye.

In the case of NAW-series materials/RR198 systems, the differences in zeta potential values tend to increase along with the MgO loading, even specific surface area of a sample

markedly decreases. As a consequence, despite the lowest specific surface area, NAW(70/30) sample (the most distinct zeta potentials difference, 62.9 mV at autogenous pH, *ca.* 10.2) demonstrates the best effect. Following this explanation, the slightly lower effectiveness during removal of BR18 from water by NAW-series samples, may be explained by smaller differences in the respective potentials.

In addition, porosity of the prepared materials also play an important role during adsorption of the dyes from water. Hence, despite comparable differences in zeta potentials, adsorption of RR198 on NAW(70/30) is much more effective than on MgO itself. Moreover, NAW-series materials containing less MgO, sorb the dye with similar efficiency like pure MgO even specific surface area of the latter is considerably lower.

In general, differences in the zeta potentials calculated for all the AW-series samples/RR198 systems do not vary much with pH, and are lower than those found for MgO-containing sorbents/RR198 systems. Despite the low difference in zeta potentials, removal of RR198 from water in the presence of acid-washed samples was quite efficient and this particularly

Figure 7. Differences of zeta potential values for: (a) sorbent/RR198 combinations and (b) sorbent/BR18 combinations.

concerns AW(70/30) material. When pore structure parameters (table 3) are considered, especially external specific surface area values, one may notice a strong increase in mesopore area with the ratio of raw materials in the initial mixture. Hence, we postulate mesopore content as a key factor in enhancing the adsorption of RR198 on MgO-free materials. Adsorption of the cationic dye (BR18) on AWs is evidently more efficient than on MgO containing samples. Considering both pore structure data and zeta potentials, it becomes clear that dissolution of MgO from NAW-series samples results in two important effects. From one side, it greatly changes pore parameters by eliminating pore-blocking effect, and from the other side, strongly changes zeta potentials and thus makes the difference between the potentials greater. It should be emphasized that great increase in specific surface area and significant increase in zeta potential differences, are the two key factors favouring BR18 removal from water. Hence the performance of AWs during BR18 removal from water is so high. The little increase in BR18 adsorption with MgO content in NAW samples (and at the same time with decrease in specific surface area) confirms some influence of the zeta potential difference (increasing with MgO content) on the process. However, the very inefficient action of MgO on BR18 confirms porosity as a factor that dominates over the difference in the potentials. In fact, while the zeta potential difference (18.1 mV at autogenous pH, *ca.* 10.2) is higher compared to NAW-series materials (from 2.3 to 17.0 mV at autogenous pH ranging from 9.3 to 9.7), the oxide shows very low specific surface area.

As shown in figures 4 and 5, carbon adsorbents prepared by us, reveal high performance during adsorption of RR198 and BR18. The dyes could be removed from water efficiently when compared to commercially available carbon (CWZ-35). This concerns especially the removal of RR198 by MgO-loaded materials. It should be noticed that acid-washed materials are also well performing during water purification from the dyes.

4. Conclusions

In this paper, MgO-loaded porous carbon materials were studied. As elucidated, a simple one-step process, may lead to a well performing sorbent. It is worthy to say that one of the raw materials (MgCO_3) used for preparations gives two-fold advantages: acts as pore-creating agent and is capable to enhance the removal of some contaminants (anionic dye) from water. As confirmed, an elimination of the oxide from the hybrid sorbent may result in obtaining a new, highly porous carbon material, with increased ability to remove another dye (cationic). It might be concluded that adsorption of ionic dyes (RR198 and BR18) on both series materials (NAW and AW) is a complex process. In the case of NAW-series sorbents, the zeta potentials of both sorbent and a dye are the key factors influencing adsorption. On the other hand, porosity has a strong impact on adsorption of the dyes on AW-series materials.

Apparently, both MgO-loaded and MgO-free adsorbents may reveal good ability to adsorb other species than the dyes studied in this work. Besides, production of the sorbents may be considered as an alternative way to dispose waste PET material.

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