



# Electrochemical degradation and extraction capability of magnesium wastes in sewage treatment

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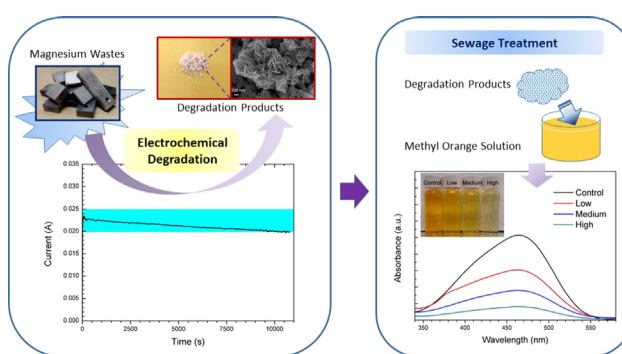
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## HIGHLIGHTS

- A facile electrochemical method is proposed based on corrosion science to degrade waste magnesium alloys.
- The degradation products are composed of mainly nanostructured magnesium hydroxide and used for sewage treatment.
- The degradation products can remove methyl orange from the simulated sewage effectively via adsorption/aggregation.

## GRAPHICAL ABSTRACT



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## ABSTRACT

Owing to the increasing use of magnesium alloys in the automotive, aerospace, electronics, and biomedical industry, more Mg-based wastes are being produced. Herein, a new process to electrochemically degrade Mg alloy wastes suitable for sewage treatment is described. Mg alloys are immersed in a sodium chloride solution and a small voltage is applied to accelerate degradation. The solid degradation products are composed of mainly nanostructured magnesium hydroxide and can be used to remove methyl orange from the simulated sewage effectively via adsorption/aggregation. This study provides insights into how to turn Mg wastes into useful forms to lessen the environmental impact.

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## 1. Introduction

Magnesium alloys have received much attention in the automotive, aerospace, electronics, and biomedical industry due to their high specific strength, light weight, and natural biodegradation [1–6]. However, as

the use of Mg-based materials continues to increase, more Mg-related wastes are generated by the industry causing potential environmental threat [7]. Therefore, in order to protect the environment and utilize natural resources more effectively, it is desirable to identify means to recycle Mg wastes.

Dissolution of magnesium in an aqueous solution proceeds by the following reaction:  $\text{Mg} + 2\text{H}_2\text{O} \rightarrow \text{Mg}^{2+} + 2\text{OH}^- + \text{H}_2 \uparrow$  [8,9]. Hydrogen, one of the products, is a clean energy source to minimize the use

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of fossil fuels, the so called low-carbon strategy. In fact, there have been recent reports on the use of Mg wastes to generate hydrogen *via* degradation in NaCl solutions and seawater added with citric acid [10,11]. Furthermore, dissolution of Mg generates  $Mg^{2+}$  and  $OH^-$  besides  $H_2$ . When those ions are saturated in the solution, magnesium hydroxide precipitates form. Magnesium hydroxide has many interesting properties such as large surface area, high endothermic decomposition temperature, and smoke suppression. It is often used in flame-retardant composites and the removal of azo-dye pigment from sewage is based on the adsorption/aggregation mechanism [12–15]. Hence, it is possible to utilize the degradation products of Mg alloy wastes in sewage treatment.

Different from stainless steels in chloride-containing aqueous solutions, magnesium alloys do not show appreciable passivation in the anodic polarization curves. Consequently, the anodic dissolution current density increases rapidly and normally has a large value even though the applied voltage is quite small [16,17]. On the other hand, the phenomenon can be exploited in that low-voltage anodic polarization can be applied to degrade Mg wastes. In this pilot study, AZ31 Mg alloy samples are immersed in a sodium chloride solution and a constant voltage of 500 mV is applied. The solid degradation product consists of mainly  $Mg(OH)_2$  which can be subsequently utilized to remove methyl orange (MO) from simulated organic dye sewage. The method enables recycling of Mg wastes to lessen the environmental impact and utilize natural resources more effectively.

## 2. Experimental details

The AZ31 Mg alloy (Mg-3 wt.%Al-1 wt.%Zn) was cut into square samples ( $20 \times 20 \times 5 \text{ mm}^3$ ), mechanically ground by 1200 grit SiC paper, ultrasonically cleaned in pure alcohol, and dried by nitrogen. The electrochemical experiments were conducted on a Zahner Zennium electrochemical workstation using the three-electrode technique. The Mg sample with an exposed surface area of  $0.5 \text{ cm}^2$  was immersed in a 3.5 wt% NaCl solution. The Ag/AgCl electrode (saturated KCl) was the reference electrode and a platinum bar was the counter electrode. A constant potential of +500 mV relative to the open potential was applied to the sample for 3 h and the degradation products were filtered and then rinsed with water twice and alcohol once. Finally, white powders were obtained after drying in air.

Field-emission scanning electron microscopy (FE-SEM; ZEISS SUPRA 55) and transmission electron microscopy (TEM; Philips CM20) were performed to examine the morphology of the samples. X-ray photoelectron spectroscopy (XPS; PHI Model 5802) was performed using Al  $K_{\alpha}$  irradiation to determine the composition and chemical states with the binding energies referenced to the C1 s line at 285 eV. X-ray diffraction (XRD; Rigaku SmartLab) with Cu  $K_{\alpha}$  radiation and selected-area electron diffraction (SAED, performed on the TEM) and field-emission high resolution transmission electron microscope (FE-HRTEM; JEOL JEM 2100F) were conducted to determine the phase and crystalline structure.

In the simulated sewage treatment, the powders were divided into three groups according to doses: low ( $0.0109 \pm 0.00106 \text{ g}$ ), medium ( $0.032 \pm 0.0007 \text{ g}$ ), and high ( $0.09223 \pm 0.00163 \text{ g}$ ). In the extraction experiments, they were ultrasonically dispersed in the simulated organic pollutants (10 mL methyl orange solution with a concentration of 10 mg/L) for 5 min and agitated for 5 h in a shaker. The solid and liquid phases were separated by centrifugation at 8000 rpm for 5 min. UV-visible spectrophotometry was adopted to determine the concentration of MO in the supernatant and the pH was measured by a pH meter.

## 3. Results and discussion

AZ31 is a common commercial magnesium alloy and it has been shown that the anodic polarization current density increases rapidly and becomes larger in small-scale polarization relative to the free

corrosion potential [18–20]. It thus means that accelerated electrochemical degradation can be carried out with high efficiency and low energy consumption. Fig. 1 depicts the current evolution of the AZ31 Mg alloy in the NaCl solution under a constant voltage. As expected, the degradation current is in the range between 20 and 25 mA within 3 h and the current evolution is relatively stable during the degradation process. Fig. 2a shows the surface morphology of the as-prepared powders with the inset showing that they are composed of aggregated nanosheets. Mg, O, C, and Ar are observed from the XPS survey spectrum (Fig. 2b) and C and Ar are contaminants. The high-resolution results (Fig. 2c) show a weak Al 2p signal indicating that there is little aluminum in the powders. This is because the Al concentration in the AZ31 Mg alloy is quite small and Al exists as mainly  $Al_xMn_y$  acting as the cathode during corrosion. Consequently, there is only a small amount of dissolved Al in the solution to take part in precipitation. The XRD pattern (Fig. 2d) shows that the as-prepared powders are mainly  $Mg(OH)_2$  and it is further corroborated by the SAED pattern in Fig. 2e. The TEM and HR-TEM images reveal the nanosheet morphology (Fig. 2e) and good crystallinity (Fig. 2f).

Fig. 3a presents the absorbance curves acquired from the methyl orange solutions with and without the added adsorbents and Fig. 3b provides the average  $C/C_0$  values calculated from the absorbance curves, where  $C_0$  is the initial concentration of methyl orange (MO) and  $C$  is the concentration of the remnant. The characteristic adsorption peak of MO decreases as more powders are added. The inset in Fig. 3a shows that the solution becomes clear after addition of the adsorbents. Fig. 3c shows that the pH of the MO solutions increases to about 10 after adsorption and there is no significant difference among the three groups (low, medium, and high). As shown in Fig. 3d, the color of the adsorbent changes from white to yellow after adsorption further corroborating that MO has been transferred from the solution into the precipitate. Therefore, it can remove MO effectively and the treated solution may be a potential candidate as an industrial acidic waste neutralizer.

Based on above experimental results, the color removal mechanism is proposed as follows. The solubility of  $Mg(OH)_2$  is given by [21,22]:  $K_{sp} = [Mg^{2+}][OH^-]^2$ , where  $K_{sp}$  is the dissociation constant of the hydroxide. Accordingly, formation of  $Mg(OH)_2$  is favored if the  $Mg^{2+}$  or  $OH^-$  concentration is increased beyond the  $K_{sp}$  value. The structure of  $Mg(OH)_2$  provides a large adsorptive surface area with a positive surface charge [23]. Hence, the  $Mg(OH)_2$  nanosheets can act as an efficient coagulant after the powders are dispersed in the MO solution. On the other hand, the magnesium hydroxide powders can dissolve in the solution. When  $Mg^{2+}$  and  $OH^-$  are fully saturated in the solution, formation of  $Mg(OH)_2$  precipitate is facilitated. Usually, the chemical reaction of  $Mg^{2+}$  conversion to  $Mg(OH)_2$  is described as follows [24]:  $Mg^{2+} + OH^- \rightleftharpoons Mg(OH)^+$ ;  $Mg(OH)^+ + H^+ \rightleftharpoons Mg^{2+} + H_2O$ ;

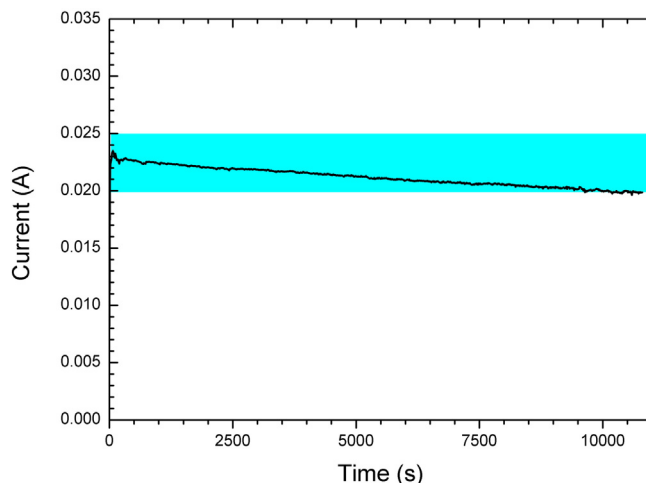


Fig. 1. Degradation currents as a function of time.

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