

Optimization of phosphate removal in anodizing aluminium wastewater

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ARTICLE INFO

Article history: Received 5 August 2005 Received in revised form 20 October 2005 Accepted 26 October 2005 <u>Available online 15 December 2005</u> <u>Keywords:</u> Magnesium oxide Phosphate removal Phosphate recovery Anodizing aluminium Magnesium phosphate

ABSTRACT

The wastewater produced after brightening and anodizing aluminium has high concentrations of phosphates and sulphates. The addition of MgO in a first physico-chemical wastewater treatment step makes the selective recovery of phosphates in the form of magnesium phosphates feasible, which may be reused as fertilizer. The proposed wastewater treatment process allows manufacturers to reduce more than 70% of the volume of the precipitate in the sedimentation reactor and more than 50% of the weight of the final disposal sludge. In this study, the use of an alternative low-grade MgO (LG-MgO) as a source of magnesium, which is cheaper than pure MgO, is investigated. The phosphate concentration and pH of the treated wastewater is controlled by the formation and precipitation of newberyite or bobierrite as a function of the magnesium source added. According to experimental data, a reaction mechanism is proposed. Although LG-MgO reacts more slowly than pure MgO and it is necessary to add 3–4 times the stoichiometric amount, this procedure has considerable economic and technical advantages.

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1. Introduction

Anodizing has greatly extended the applications of aluminium in products and uses where the metal might otherwise not be utilized. The process provides a much thicker oxide coating, several microns thick if required, improving the corrosion resistance, abrasion resistance, and overall appearance of aluminium. Adequate cleaning and rinsing are the first required process operations. Deoxidizing would normally be the next step. Using an acid solution and a somewhat elevated temperature, the purpose of deoxidizing is to remove non-uniform oxide films present on the surface of aluminium and other contaminants not dissolved by the soak cleaner. Deoxidizers are typically mixtures of mineral acids. The mixture selectively attacks the metal surface protrusions, resulting in an overall levelling effect. After that, the aluminium follows either an etching process, in order to

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remove the natural shine, providing a soft, matte, texture appearance, or a brightening process. For brightening, a mixture of acid solutions, nitric (3–6%), sulphuric (10–20%) and phosphoric (80–85%), are usually used prior to anodizing, resulting in a mirror-shiny surface. Anodizing is usually carried out in a nominal 15–17% sulphuric acid solution at 21–27 °C. Finally, dyeing (if necessary) and sealing are the last final process steps.

As a result of the process described, the wastewater obtained after the production in bulk of anodized aluminium contains high amounts of phosphates and sulphates, nitrates and, to a somewhat less degree, aluminium, as well as a very low pH. In order to be able to reuse these waters or to discharge them properly, the ions must be removed in a physico-chemical treatment plant.

Moreover, phosphate is recognized as being one of the resources that will be scarce in the near future. So, it is of

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^{0043-1354/\$ -} see front matter @ 2005 Elsevier Ltd. All rights reserved. doi:10.1016/j.watres.2005.10.033

value to recover most of the phosphate from the liquid waste. The removal of phosphorus has been widely studied and, at present, two effective and reliable methods have been established: chemical precipitation and biological removal (Morse et al., 1993). In most chemical treatments, the phosphorus can be removed from sewage by precipitating it via a metal salt, e.g., iron, aluminium and mainly calcium salts (Strickland, 1998; Donnert and Salecker, 1999; Angel, 1999; House, 1999). Phosphorus removal via biological treatment has been developed over the course of the last 20 years and is now beginning to compete with the more conventional physico-chemical approach of precipitation with metal salts, mainly in municipal wastewater and animal manure treatments (Woods et al., 1999; Greaves et al., 1999). Both chemical and biological removal methods allow phosphorus to be recycled as a sustainable product for use as a raw material in either industrial or agricultural applications (Durrant et al., 1999; Stratful et al., 1999). Thus, phosphorus removal could be made more sustainable if combined with recovery.

Nevertheless, as a consequence of the fact that magnesium phosphates have a very low solubility ($pK_{sp} = 25.2$), it is possible to use magnesium salts to remove the phosphates in wastewater instead of the commonly used calcium salts, mainly lime. In this case, MgO seems to be the most appropriate raw material used for the physico-chemical wastewater treatment. There are many advantages in using MgO as raw material: MgO has minimal environmental impact, has a low solubility, and is essential for plant, animal and human growth; MgO also has a high alkalinity (more than other alkalis), which helps to neutralize acids and precipitate metals requiring less Mg(OH)₂ to neutralize the same amount of acid; Mg(OH)₂ is a weak base and its dissolution is not exothermic, thus being able to reach a maximum pH of 9, in accordance with the Clean Water Act basic limits; and the sludge formed by the reaction is conducive to crystal growth and is not light, fragile or gelatinous like that formed by other alkalis (Teringo III, 1987).

Furthermore, in wastewaters with high phosphate and sulphate contents like those obtained in the anodizing aluminium process, while the use of lime precipitates phosphates and sulphates together, the use of MgO allows selective precipitation of the phosphate without the presence of sulphates, since magnesium sulphate is very soluble (about 250 gL^{-1}). These facts facilitate the recovery of phosphates, which may be used directly as a fertilizer or a raw material in industrial applications.

While the use of MgO for the removal and recovery of phosphates would be technically feasible for the treatment of high-strength wastewater, it is not adopted economically because of the high cost of magnesium compounds (Giesen, 1999), i.e., chemically reactive Mg(OH)₂ is eight to ten times more expensive than a similar quality of calcium hydroxide. However, it is possible to use other sources of magnesium that are more economically feasible. Thus, low-grade MgO (LG-MgO) may be used for the removal of phosphates (Chimenos et al., 2003).

In the present study, experiments to remove the phosphates from anodizing aluminium wastewaters are performed using LG-MgO. The physico-chemical wastewater treatment studied at laboratory scale is depicted in Fig. 1.

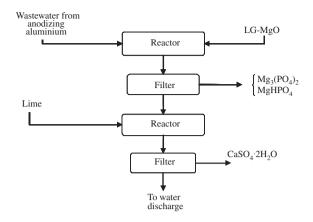


Figure 1 – Flowsheet of the physico-chemical treatment of wastewater from anodizing aluminium.

The process combines two consecutive steps. In the first one, phosphorus is removed by forming a precipitate of magnesium phosphate, while the sulphate remains in solution. In the second step, after filtering the sludge formed in the previous step, lime is added to precipitate sulphate as gypsum. The recovery of magnesium phosphates, which are suitable to reuse, reduces the volume and weight of the final disposable sludge. The objective of this study is to determine the effectiveness of LG-MgO as a source of magnesium and the optimum parameters at laboratory scale needed for a further design of a physico-chemical pilot plant.

2. Methods and materials

This study was carried out with wastewater from the anodized processing of aluminium to produce spray pumps that are used in cosmetic and pharmaceutical markets. Nowadays, the process generates 4 m^3 of wastewater per hour and the physico-chemical treatment with lime produces $7-8 \text{ ton } \text{day}^{-1}$ of sludge that, in accordance with local legislation, must be disposed of as a special residue in a second class landfill. The wastewaters generated from the different steps are mixed prior to the physico-chemical treatment. A wastewater sample of 10L of this mixture was used to perform laboratory trials. The chemical bulk analysis of an aliquot is shown in Table 1.

Analytical grade MgO and LG-MgO were used as a source of magnesium. The LG-MgO used is produced and sold by Magnesitas Navarras, S.A. located in Navarra (Spain). It comes from the calcination in rotary kiln at 1100 °C of natural magnesite. The product is collected as cyclone dusts in the fabric filters from the air pollution control system. The initial price of this low-grade product is close to that of the calcium hydroxide commonly used in wastewater treatment plants. The composition and other physical parameters are also shown in Table 1.

The experiment trials were performed using a flocculation tester that consists of six 400 mL beakers that are agitated simultaneously at 200 rpm. All experiments were carried out at a room temperature of 25 °C. Different solid/liquid (S/L) ratios, from 8 to 10 gL^{-1} for LG-MgO additions and 5.7 to

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