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Development of new materials from waste electrical and electronic equipment: Characterization and catalytic application

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ABSTRACT

Wastes of electrical and electronic equipment (WEEE) represent an important environmental problem, since its composition includes heavy metals and organic compounds used as flame-retardants. Thermal treatments have been considered efficient processes on removal of these compounds, producing carbonaceous structures, which, together with the ceramic components of the WEEE (*i.e.* silica and alumina), works as support material for the metals. This mixture, associated with the metals present in WEEE, represents promising systems with potential for catalytic application. In this work, WEEE was thermally modified to generate materials that were extensively characterized. Raman spectrum for WEEE after thermal treatment showed two carbon associated bands. SEM images showed a metal nanoparticles distribution over a polymeric and ceramic support. After characterization, WEEE materials were applied in ethanol steam reforming reaction. The system obtained at higher temperature (800 °C) exhibited the best activity, since it leads to high conversions (85%), hydrogen yield (30%) and H_2/CO ratio (3,6) at 750 °C.

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1. Introduction

Over the last fifteen years, quantities of wastes of electrical and electronic equipment (WEEE) have increased rapidly, mainly due the modernization and technological development (Babu et al., 2007). These wastes include refrigerators, dishwasher, cell phones, computers, televisions, etc., and its composition depends strongly on the type and age of the equipment. In general, WEEE is composed of metals (40%), plastic (30%) and refractory oxides (30%), as shown in Fig. 1 (Sodhi and Reimer, 2001; Gramatyka et al., 2007; Li et al., 2007).

Typical metal constitution includes copper (20%), iron (8%) tin (4%), nickel (2%), lead (2%), zinc (1%), silver (0.02%), gold (0.1%) and palladium (0.005%) Sum, 1991. The most important plastic components are polyethylene, polypropylene, polyesters and polycarbonates (Sodhi and Reimer, 2001).

Some organic compounds as tetrabromobisphenol A (TBBPA), polychlorinated biphenyls (PCB), hexabromocyclododecane (HBCD), polybrominated diphenyl ether (PBDE) are extensively used in electrical and electronic industry to produce flame resistant resins (Darnerud, 2003). Some flame retardants, *e.g.* PBDE's, were classified as persistent organic pollutants (POP) due to their dangerousness and problems caused in the treatment and recycling of WEEE (Wang and Zhang, 2012; EEA, 2010).

Thermal treatments have been considered efficient to WEEE treatment (Zhang and Zhang, 2012). After heating, a carbonaceous structure is produced, which could work as support with silica and alumina, present in these wastes (Rournanie et al., 2008). This mixture, associated with the metals present in WEEE, as Fe, Cu, Ni, Pd and Pt represent promising systems with potential for catalytic application (Furtado et al., 2009; Thomas, 1996).

Many metals present in WEEE are commonly applied in several different catalytic processes, such as ethanol steam reforming (ESR), an important route to produce H_2, an ideal source of clean and sustainable energy, with high efficiency in fuel cells. Three main catalytic pathways are used to produce hydrogen from ethanol: steam reforming, partial oxidation, and its combination, autothermal reforming.(Divins et al., 2015) The use of ethanol as fuel sources for hydrogen production has attracted much attention, owing to its easy handling, wide distribution around the world, safe transportation and storage, low toxicity and high volumetric energy density, besides it can be produced from several biomass sources.(Kourtelesis et al., 2015; Zhao et al., 2016)

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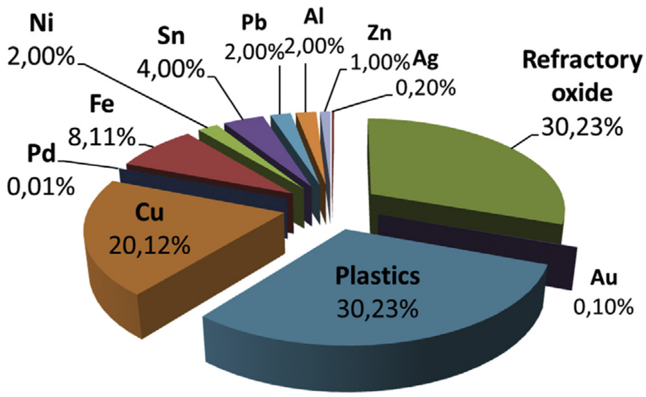

Fig. 1. Typical composition of WEEE (Adapted from Gramatyka et al. (2007)).

In these reactions, ethanol and water react, generating hydrogen and carbon dioxide in a 3:1 ratio (Rosmaninho et al., 2012) (Eq. (1)).

$$C_2H_5OH + 3H_2O \rightarrow 6H_2 + 2CO_2 \quad (1)$$

Despite of the simplicity of Eq. (1), the ethanol steam reforming occurs through a very complicate pathway including different simultaneous reactions, as water-gas shift (Eq. (2)), ethanol decomposition (Eq (3)), ethanol dehydrogenation (Eq. (4)), ethanol dehydration (Eq. (5)), acetaldehyde steam reforming (Eq. (6)) and acetaldehyde decomposition (Eq. (7)) Ito and Tomishige, 2010; Buitrago-Sierra et al., 2012; Yun et al., 2012.

$$CO + H_2O \rightarrow H_2 + CO_2 \quad (2)$$

$$C_2H_5OH \rightarrow CO + CH_4 + H_2 \quad (3)$$

$$C_2H_5OH \rightarrow CH_3CHO + H_2 \quad (4)$$

$$C_2H_5OH \rightarrow C_2H_4 + H_2O \quad (5)$$

$$CH_3CHO + 3H_2O \rightarrow 5H_2 + 2CO_2 \quad (6)$$

$$CH_3CHO \rightarrow CH_4 + CO \quad (7)$$

Ethanol steam reform produces high yields of hydrogen at elevated temperatures. A variety of materials have been shown high activity for these reaction (Oliveira et al., 2013), mostly systems based on Zn, Mg, and V oxides (López et al., 2012), noble metals, as Pd, Pt, Rh e Ru (Domínguez et al., 2012; Idriss et al., 2008; Deluga et al., 2004; Frusteri and Freni, 2007) and base metals (Navarro et al., 2007). On the other hand, recently, alternative processes operating at lower temperatures have been investigated. Several supported metals, including Ni, Co, Cu, Ir, Pt, Rh, Pd and Ru exhibit good activity at low temperature (300–500 °C) Torres et al., 2007; Casanovas et al., 2010; Comas et al., 2004. However, these materials undergo severe deactivation due to carbon deposition, which can be avoided using excess of water during the reaction. Furthermore, supports, such as Al_2O_3, ZnO, MgO, La_2O_3 and CeO_2, have been extensively studied (Divins et al., 2015; Buitrago-Sierra et al., 2012; López et al., 2012; Torres et al., 2007; Palmeri et al., 2007; Resini et al., 2007; Liao et al., 2011; Profeti et al., 2009; Garbarino et al., 2015; Dancini-Pontes et al., 2015; Hedayati et al., 2015; Rosmaninho et al., 2010; Andreev et al., 2014).

In this work, wastes of electrical and electronic equipment were thermally modified, producing new materials with high catalytic power, potently applicable in several processes, as ethanol steam reforming reactions.

2. Experimental

2.1. Preparation of WEEE

Wastes of electrical and electronic equipment obtained from discarded printed circuit boards (PCB) were first cut in an industrial slicer machine and sieved to 60 mesh. In a second step, the sieved material was grinded on a ring mill for 5 min, until a fine powder was obtained.

The grinded WEEE was submitted to thermal treatment in order to remove flame retardant compounds and organic materials. According Oleszek et al. (2013), brominated flame retardants (BFRs) such as tetrabromobisphenol A (TBBPA) start their decomposition at 220 °C and, around 320 °C, 95% of sample was decomposed. In presence of metal, BFRs thermal decomposition occur at similar temperatures, which explains the choice of treatment temperatures in this work.

About 3.0 g of WEEE powder was packed in a quartz tubular reactor and heated under air atmosphere at 10 °C min^{-1} from 25 to three different temperatures, 400, 600 and 800 °C. The final temperature was maintained for 1 h.

2.2. Characterization

The materials prepared from WEEE and the raw waste were characterized by X-ray diffraction (Philips-1830 equipment using Cu Kα radiation and scanning from 2° to 75° at scan rate of 4° min^{-1}), X-ray fluorescence (Shimadzu EDX-720 equipment), Mössbauer spectroscopy (CMTE model MA250, ^{57}Co/Rh source at room temperature, α-Fe as reference), scanning electron microscopy (FEG – Quanta 200 FEI), Raman spectroscopy (Brüker Senterra, excitation laser with wavelength of 633 nm) and specific area determination by nitrogen adsorption using B.E.T. method (Autosorb 1 Quantachrome instrument).

Thermal analyses (TG/DTG/DTA) were carried out in a DTG 60H Shimadzu under air flow of 50 mL min^{-1} and heating rate 10 °C min^{-1}. Thermo-gravimetric analysis coupled to mass spectrometry (TGA-MS) were done in a Netzsch STA449 thermo balance coupled to a QMS 403C mass analyzer, with heating rate of 10 °C min^{-1} under argon flow (20 mL min^{-1}).

Al, Cu, Fe, Ni, Sn, Pb and Zn approximated amounts present in the waste were determined by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) analysis, on a Perkin-Elmer Optima 3300-DV equipment. Before the analysis, the sample was digested for 24 h in HCl-HNO_3 3:1 solution and then, filtered and washed with water. Semi-quantitative analysis by X-ray fluorescence spectroscopy (XRF), on a Shimadzu EDX-720 equipment, were also carried out to identify other elements present in the WEEE.

The TPR analyses were carried out on a Quantachrome Chem-BET3000 (10% H_2 in N_2 mixture, flow of 22 mL min^{-1}, heating rate of 10 °C min^{-1} up to 900 °C).

2.3. Catalytic reactions

Ethanol steam reforming were performed for the raw waste (WEEE) and the material thermally treated at 800 °C (WEEE-800), as well these materials after reduction pre-treatment (WEEE Red and WEEE-800 Red). The reduction treatment was carried out in hydrogen atmosphere (20% H_2/N_2, 62.5 mL min^{-1}) up to 800 °C (10 °C min^{-1}) for 10 min. For ethanol steam reforming reactions, the powder (100 mg) was packed in a quartz tubular reactor and heated in a conventional gas-flow system with a fixed catalyst bed.

On the heat up reaction, the samples were thermally treated from 25 to 900 °C (at 5 °C min^{-1}) for 1 h. A peristaltic pump was

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