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Hydrogen production by steam reforming of acetic acid using hydrotalcite type precursors

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ABSTRACT

Acetic acid can be used as a model compound in the study of the steam reforming of the hydrophilic phase of bio-oil. In order to perform acetic acid reforming reactions, low cost catalysts with high catalytic activity and selectivity are required. Thus, Ni–Mg–Al hydrotalcite derived catalysts emerge as a good alternative. In this work, four Ni–Mg–Al hydrotalcite type precursors were prepared, with different Ni/Mg molar ratios (0.3, 0.4, 0.5, and 0.6). Various characterization tests have been performed for the prepared hydrotalcite type precursors. The stability of the catalytic activity was evaluated during 24 h and the used samples were submitted to a thermogravimetric analysis to evaluate carbon accumulation. Characterization tests proved that Ni/Mg ratios had a direct influence in the reducibility of the precursors. The Temperature-programmed surface reaction analysis showed that the steam reforming of acetic acid occurred majorly when the temperature reached 873 K. Stability test showed that precursors 04NiMg and 05NiMg had the best catalytic performance. It was also seen that sample 06NiMg suffered from deactivation by carbon deposition, which was confirmed by the thermogravimetric analysis.

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Introduction

Hydrogen technologies have several interesting advantages that can be explored. Hydrogen has the highest energy density of all known energy sources, about 2.75 times higher than petroleum derivatives [1] and its combustion does not generate greenhouse gases as CO and CO₂. Industrially, hydrogen is generally obtained from methane reforming [2], but it can also be produced from renewable materials. Actually, the use of hydrogen and of other renewable energy

sources might be crucial in the near future, since the global power consumption overpassed 13 TW and it is still increasing rapidly [3]. In recent years, a lot of research effort has been done studying the use of biomass in pyrolysis or gasification processes to obtain bio-oil as a possible renewable energy source [2,4–6]. The aqueous fraction of bio-oil is mainly composed by water and organic oxygenates like acids, ketones and aldehydes [7,8]. Unlike the hydrophobic fraction, the aqueous fraction has a lack of utility and most of the time it is destined for disposal. Therefore, it would be advantageous to

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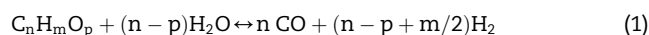
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Nomenclature

AcAc	Acetic Acid
LCF	Linear Combination Fitting
RWGS	Reverse Water Gas Shift
SRAA	Steam Reforming of Acetic Acid
TGA	Thermogravimetric Analysis
TPR	Temperature Programmed Reduction
TPSR	Temperature Programmed surface reaction
WGS	Water Gas Shift
XANES	X-ray Absorption Near Edge Structure
XRD	X-ray Diffraction

use the aqueous fraction of bio-oil to produce hydrogen [9,10]. However, due to the great variety of compounds in the aqueous fraction it is difficult to set optimum operational conditions aiming the production of hydrogen. One possible solution to this issue is to use a model molecule to represent the whole aqueous fraction [11]. Since acetic acid (AcAc) is the most abundant organic component of the aqueous fraction of bio-oil (up to 12% wt), it is interesting to use it as a model molecule on steam reforming reactions to produce hydrogen [12–14].

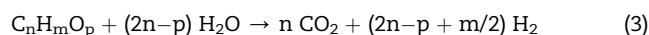
It has been reported [4,10,15,16] that the steam reforming of organic oxygenates can be written as follows in Eq. (1):



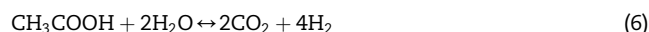
It is known that the water gas shift (WGS) reaction also occurs simultaneously with the steam reforming reaction. The WGS reaction (Eq. (2)) is responsible for part of the conversion of carbon monoxide that was generated in the steam reforming into carbon dioxide.



Eqs. (1) and (2) can be combined to form a global equation as follows:



Mohanty et al. [15] and Zhang et al. [17] have shown the application of Eqs. (1)–(3) in the steam reforming of acetic acid (SRAA). The reactions involving acetic acid can be seen in Eqs. (4), (5) and (6):



To conduct the SRAA reaction, catalysts with desirable characteristics are necessary. It is interesting to choose metals that have low cost, relatively high activity and selectivity for the desirable products [18]. Basagiannis and Verykios [13] investigated several metals (Ru, Rh, Ni, Pd and Pt) supported on Al_2O_3 , CeO_2/Al_2O_3 , La_2O_3/Al_2O_3 and MgO/Al_2O_3 for SRAA. The results showed that, among all metals, Ni, Rh and Ru presented the best performances. The addition of cerium,

lanthanum or magnesium oxides to the alumina support improved significantly the activity and stability of the catalysts. Besides, the presence of La_2O_3 and MgO led to an expressive reduction on carbon deposition rates. Although many times noble metals based catalysts present a better catalytic performances, their costs are still very high. Thus, nickel based catalysts seem to be a more reasonable choice. Nickel favors C–C and C–H bond cleavage which is also required in a steam reforming process [19]. However, it is important to take in consideration that nickel catalysts may suffer deactivation by carbon accumulation as reported in literature [20,21]. Among all types of nickel catalysts, the use Ni based hydrotalcites as catalyst precursors could be a good option in the SRAA to produce hydrogen. The capacity to generate uniform mixed oxides with high superficial area are certainly the major advantages of their application in the SRAA reaction [22,23]. Bimbela et al. [24] have studied the effect of copper and magnesium contents on co-precipitated Ni–Al precursors for the catalytic reforming of model compounds (acetic acid, acetol and butanol). The authors verified that the sample containing 5 wt% of Cu presented the best catalytic performance in terms of H_2 yield. Copper inhibited the formation coke that could lead to catalyst deactivation in acetic acid steam reforming with a steam-to-carbon molar ratio (S/C) of 5.6. On the other hand, such a positive effect of copper was not observed in acetic acid steam reforming with S/C = 14.7. Montañez et al. [23] studied steam reforming of ethanol using hydrotalcite-like precursors Ni–Mg–Al. The results showed that it was possible to obtain catalyst precursors that led to good selectivity for H_2 production by co-precipitation and urea hydrolysis. Regardless of the preparation methodology and the fact that some carbon deposits were observed during stability tests, all samples with nickel loadings between 10 and 20% had 100% ethanol conversion at 823 K and maintained stable activity. Guil-Lopez et al. [25] evaluated steam reforming of acetone also using Ni–Mg–Al hydrotalcite-like precursors and nickel catalysts supported on MgO/Al_2O_3 catalysts. They showed that one of the catalysts derived from hydrotalcite-type structure (26 wt% of Ni) presented the lowest methane formation, which was linked to higher reforming capacity, whereas high methane formation was related to high acetone decomposition activity. The authors pointed out that the hydrotalcite synthesis leads to precursors with better reforming activity rather than decomposition of the acetone molecule. Considering that hydrotalcite decomposition may be an interesting path to obtain these catalysts, the objective of this work was to evaluate Ni–Mg–Al hydrotalcite-like materials with different nickel loadings as catalyst precursors for the steam reforming of acetic acid to produce hydrogen.

Methodology

Catalyst preparation

Four hydrotalcite-like precursors Ni–Mg–Al were prepared through co-precipitation method in constant pH (pH = 10.0 ± 0.5). The molar proportion of bivalent metals Ni^{2+}/Mg^{2+} were set to 0.3, 0.4, 0.5 and 0.6, which generated samples

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