Catalytic activity and stability of sulfonic-functionalized UiO-66 and MIL-101 materials in friedel-crafts acylation reaction

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ABSTRACT

Sulfonic-containing UiO-66 and MIL-101 MOF materials, prepared by direct synthesis with a sulfonic acid-including benzene dicarboxylate (SO_3H-BDC) linker, have been evaluated as acid catalysts in Friedel–Crafts acylation of anisole with acetic anhydride. The catalytic activity of these materials was compared to other conventional acidic sulfonic heterogeneous catalysts, such as commercial Nafion-SAC-13 and Amberlyst-15. The catalytic performance of MOF materials was significantly dependent on their textural properties and the availability of sulphonic acid groups. MIL-101-SO_3H material displayed a remarkable anisole conversion and specific activity per sulfonic acid centre due to its open structure and multimodal pore size distribution. The inherent properties of MIL-101-SO_3H material allowed a more sustainable catalyst regeneration than those used for conventional heterogeneous catalysts due to the deposition of reagents and products, in particular poly-acetylated compounds. MIL-101-SO_3H proved an easy recovery and reusability in successive runs without any loss of activity. These promising results evidenced the potential of MIL-101-SO_3H as an alternative catalyst for acid-catalyzed reactions.

1. Introduction

The replacement of homogeneous catalysis by heterogeneous systems is a relevant challenge in current industrial chemistry, particularly in traditional acid-catalyzed chemical processes [1]. A high number of liquid phase reactions of chemical manufacturing processes use acid catalysts, more commonly homogeneous conventional acids such as $AlCl_3$, HF, H_2SO_4, etc. The use of these types of catalysts is associated with unfriendly processes for the environment, like the generation of a large volume of corrosive and toxic wastes [2,3]. These acids are generally soluble in the reaction medium and decompose after reaction, being impossible to reuse them. On the other side, heterogeneous catalyst systems also present several drawbacks like poisoning, low active site accessibility or leaching of catalytic groups.

In the last years, many different types of materials have been used as heterogeneous acid catalysts. These materials can be classified concerning their Brønsted /Lewis acidity, the strength and number of acid centres and the matrix support, which is particularly important to achieve a successful heterogeneous catalyst [4]. Thus, the chemical and thermal stability, as well as the catalytic activity, are associated with the structure and composition of the support [5]. Porous solids are normally used as heterogeneous catalysts due to their high surface area and wide range of pore size, which facilitate the substrate accessibility to the active sites [6]. Zeolitic microporous materials were one the first materials used at an industrial scale, in particular, in fluid catalytic cracking (FCC) of petroleum crude oils [7]. Both Brønsted and Lewis acid sites occur in zeolites and their internal structure provides a high shape selectivity for reactants and/or products. For these reasons, zeolites are commonly used as selective acid heterogeneous catalysts in refining, petrochemical, and fine chemistry industries. Functionalized ordered mesoporous silica materials have also been widely used as acid heterogeneous catalysts (Brønsted and Lewis) due to the easy incorporation of a high number of different acid functionalities onto the support [4, 8–10], as well as the possibility of tuning the pore size to get high surface areas and pore volumes.

More recently, metal-organic frameworks (MOFs) have shown also
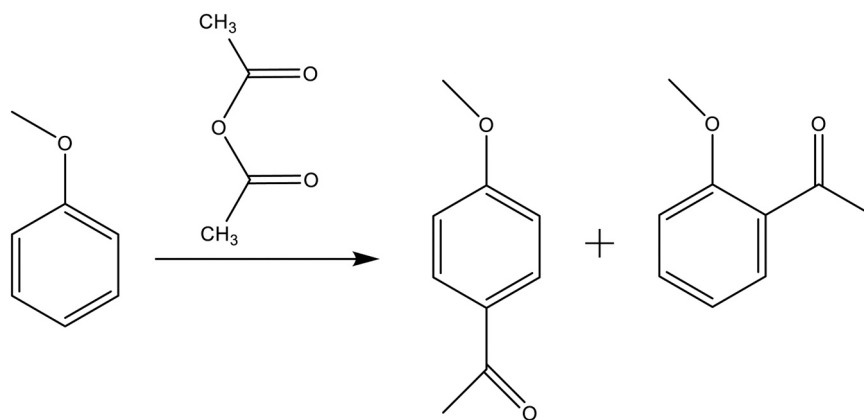
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Scheme 1. Simplified reaction scheme for the acylation of anisole with acetic anhydride.

great potential as heterogeneous catalysts. MOFs are an exciting class of crystalline materials built from metal ions (or metallic clusters) with bridging organic linkers [11,12]. Due to their large surface area, adjustable pore structure and active centres, MOFs have received much attention in the catalytic field [13–15]. Particularly, UiO-66 (Universitetet i Oslo) and MIL-101 (Materiaux de l'Institut Lavoisier) families are considered promising materials in heterogeneous catalysis [16–19] because of their excellent porosity, chemical stability and tunability of structural metals and functionalities. The UiO-66 structure consists of $Zr_6O_4(OH)_4$ clusters, which contain six-centred Zr cations and eight $\mu3$-O bridges, connected by terephthalate ligands, resulting in a structure with *fcu* topology and microporous cages [18]. Meanwhile, the MIL-101 crystal structure is a metal(III) carboxylate built from trimers of metal octahedra sharing a common oxygen and are also linked by terephthalate ligands [20]. It should be noted that MIL-101 type materials possess mesoporous cages and, by appropriate thermal treatment, coordinatively unsaturated cationic sites (CUSs), which can act as catalytic active sites [21,22]. Although there has been much progress in Lewis acidic MOF-based catalysts, just scarce studies exist on the use of Brønsted acidic MOFs with moderate and superacidic strength as heterogeneous catalysts for organic reactions [23,24]. Among the different approaches developed to introduce Brønsted acidity into metal-organic frameworks, the preparation of sulfonic acid-functionalized MOFs is a promising alternative. For instance, sulfonic acid functional groups were successfully incorporated into Al-MIL-53-NH2 by post-synthetic modification of the amino groups for the [4 + 2] cycloaddition of 2-vinyl-substituted phenols [25]. Following also a post-synthetic procedure, sulfonic acid functionalized IRMOF-3, MIL-101(Cr) and MIL-100(Fe) were prepared and applied for the synthesis of acrylonitriles [26], acetals [27] and biodiesel [28], respectively. On the other hand, the potential sulfonic acid-functionalized UiO-66, prepared by direct synthesis with sulfonic acid-containing benzene dicarboxylate (BDC) linker, has been studied and tested in the obtention of γ-valerolactone [29,30] and acylation of p-Xylene with benzoyl chloride [31]. Another example of sulfonic acid-functionalized MOF by in situ preparation using SO3H-BDC linker is the MIL-101-SO3H, which has shown excellent features to catalyze the liquid-phase Beckmann [32] rearrangement and Hantzsch reaction [33].

Friedel-Crafts acylation of aromatic compounds is an important synthetic route to obtain aromatic ketones mainly used as intermediates in the synthesis of fragrances, insecticides and pharmaceutical products [34]. These electrophilic acylations are catalyzed by Lewis or Brønsted acids. In recent years, numerous studies have been reported using acid heterogeneous catalysts as zeolites [35], metal oxides [36] and functionalized ordered mesoporous silica materials [37], but MOFs are not so frequently used in this reaction [31,38,39]'.

MOF materials were catalytically active due to the unsaturated Lewis acid sites of metal centres [38,39] but sulfonated UiO-66 demonstrated

an enhanced acid character of the catalyst through the introduction of additional Brønsted acid sites [31]. An important drawback associated with the use of heterogeneous acid systems in Friedel-Crafts acylation is a quick deactivation of the catalytic system due to the formation of poly-acetylated products that are strongly adsorbed on the acid centres.

In this work, we report the potential of sulfonic acid-functionalized UiO-66 and MIL-101 materials, prepared by direct synthesis with a sulfonic acid-containing benzene dicarboxylate (SO3H-BDC) linker, as heterogeneous catalysts in the Friedel–Crafts acylation reaction of anisole using acetic anhydride as acylation agent. The catalytic activity of these materials has been compared to other conventional acidic sulfonic heterogeneous catalysts, such as commercial Nafion-SAC-13 and Amberlyst-15.

2. Experimental section

2.1. Chemicals

Methanol (≥99.8%), N,N′-dimethylacetamide (DMA, ≥99.5%), ethanol (≥99.8%), and hydrochloric acid (HCl, 37.0%) were obtained from Scharlau. Monosodium 2-sulfoterephthalic acid (BDC-SO3Na, >98%) was procured from Tokyo Chemical Industry. Zirconyl chloride octahydrate (ZrOCl2·H2O, ≥99%) was acquired from Merck. Chromium trioxide (CrO3, ≥99%) and formic acid (≥99%) were obtained from Sigma-Aldrich. Nafion-SAC-13 composite and Amberlyst-15 resin were also supplied by Sigma-Aldrich.

2.2. Synthesis of UiO-66-SO3H and MIL-101-SO3H

UiO-66-SO3H and MIL-101-SO3H were synthesized by a solvothermal route following similar procedures previously described [40,41]. Briefly, UiO-66-SO3H was prepared by mixing 1 g of ZrOCl2.8 H2O and 0.83 g of BDC-SO3Na in a solution of 11.7 mL of formic acid and 40 mL of DMA. The mixture was stirred for 30 min, transferred to a 100 mL Teflon-lined stainless-steel autoclave and heated at 423 K for 24 h. The resulting white solid was collected by centrifugation, washed with ethanol and dried under vacuum. For the preparation of MIL-101-SO3H, 3.35 g of BDC-SO3Na and 1.25 g of CrO3 were mixed in 50 mL of water. Then, 0.91 g of concentrated HCl were added to the mixture with constant stirring. The mixture was introduced to a 100 mL autoclave and heated at 453 K for 6 days. The reaction product was separated by filtration and washed with deionized water and methanol. To purify the green solid, it was washed with 50 mL of ethanol at 373 K for 24 h.

2.3. Characterization

X-ray diffraction (XRD) patterns were obtained using Cu K$_\alpha$ radiation on a Bruker D8 Advance diffractometer. Nitrogen adsorption isotherms
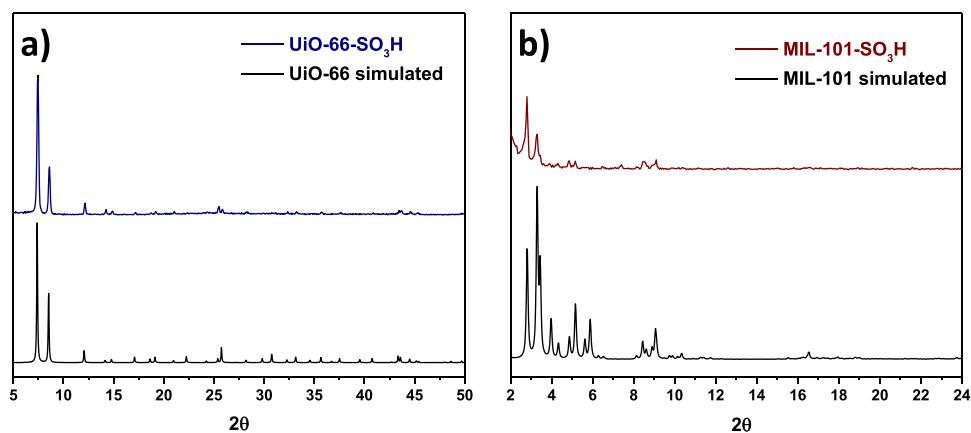
Fig. 1. Powder X-ray diffraction patterns of simulated and experimental (a) UiO-66-SO₃H and (b) MIL-101-SO₃H. Simulated diffractograms of UiO-66 and MIL-101 structures were obtained from their crystallographic data [42,43].

were measured at 77 K by using a TriStar II (Micromeritics) gas adsorption analyzer. The samples were previously outgassed at 413 K overnight. The isotherms were analyzed by using the Brunauer-Emmett-Teller (BET) method to determine the specific surface area and the two-dimensional non-local density functional theory (2D-NLDFT) model for the determination of the pore size distribution. Simultaneous thermogravimetry and derivative thermogravimetric analyses (TGA/DTG) were carried out under a nitrogen atmosphere with an airflow of 100 mL min^{-1} at a heating rate of 5 °C/min up to 800 °C, using a TA Instruments SDT 2860 apparatus. For IR experiments, thin self-supported wafers of the samples were prepared and degassed inside an IR cell under dynamic vacuum at 453 K for 7 h. After this thermal treatment, FTIR spectra of the MOF samples, before and after carbon monoxide adsorption, were recorded using a Bruker Vertex 80 v spectrophotometer working at 3 cm^{-1} resolution to study the acid sites of the samples. The presence of S in the reaction media was determined by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) using a Varian Vista AX Instrument. Elemental analyses of the catalysts for C, H, N, and S were carried out with a PerkinElmer 240 C elemental analyzer.

2.4. Reaction procedure

In order to evaluate the catalytic performance of UiO-66-SO₃H and MIL-101-SO₃H materials, the acylation of anisole with acetic anhydride to form methoxyacetophenones (MAPs) was studied as a reference acid-catalyzed reaction (Scheme 1). All the catalytic experiments were carried out under N₂ atmosphere in a round bottom flask placed in a silicone bath. Reactants and the catalyst were charged at room temperature, and then heated up to 423 K. Equimolar anisole/acetic anhydride molar ratio, 1.25 wt% of catalyst relative to anisole mass and absence of solvent were used. The stirring was fixed at 500 rpm to avoid diffusional limitations. Note that MOF materials employed as catalysts were activated under vacuum at 423 K for 2 h before being used in the reaction. Aliquots were withdrawn at selected reaction times of 1 and 5 h. Anisole and o- and p-MAPs were quantified by gas chromatography, using a GC-3900 Varian chromatograph equipped with a CPSIL 8 CB capillary column (30 m × 0.25 mm, film thickness 0.25 μm) and a flame ionization detector (FID). Sulfolane was used as an internal standard and all samples were analyzed twice.
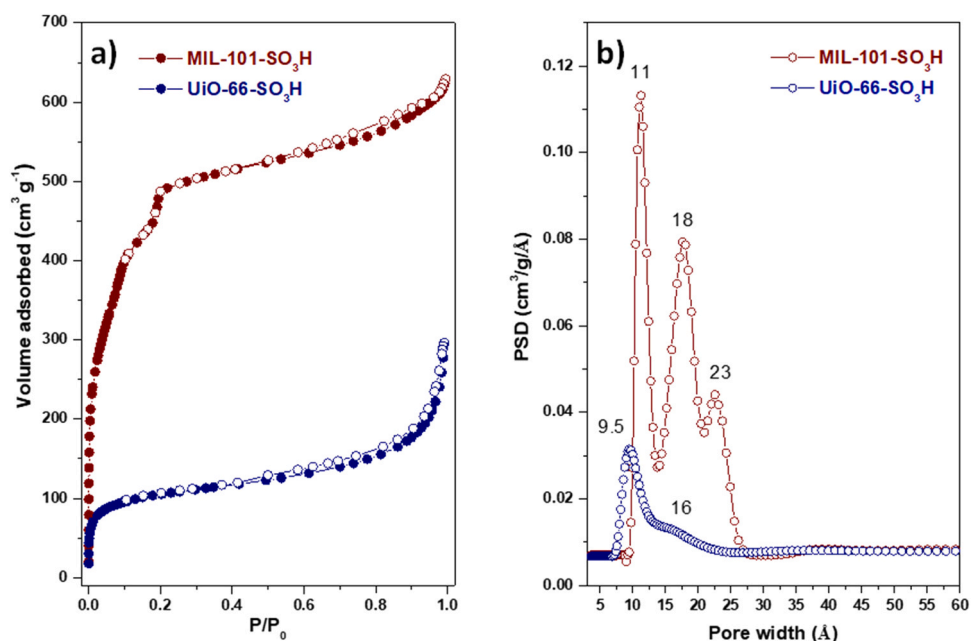

Fig. 2. (a) Nitrogen adsorption-desorption isotherms and (b) pore size distribution of MIL-101-SO₃H and UiO-66-SO₃H.

Table 1
Physicochemical properties sulfonic acid-MOF and reference catalysts.

Sample	S_{BET} (m^2 g^{-1})	V_p (cm^3 g^{-1})	Pore width (Å)	(mmol S/g)[a]
UiO-66-SO$_3$H	390	0.4	9.5 / 16	0.91
MIL-101	1986	1.19	12/ 18 / 23	–
MIL-101-SO$_3$H	1506	0.95	11/ 18 / 23	0.68
SAC-13	200	0.43	100	0.13
Amberlyst-15	50	0.40	300	4.80

[a] Sulfur content calculated from elemental analysis.

Table 2
Catalytic performance of acid sulfonic acid-MOF and reference catalysts.

Sample	Anisole conversion		Specific Activity[a]	
	1 h	5 h	1 h	5 h
UiO-66-SO$_3$H	0.3	0.5	3.3	5.5
MIL-101	0.4	0.9	–	–
MIL-101-SO$_3$H	9.7	14.8	81.5	113.4
Amberlyst-15	11.5	28	13.6	34.6
SAC-13	8.5	13.1	399.1	612.3

[a] Specific activity as reacted mmol of anisole per mmol of sufonic acid site.

3. Results

3.1. Characterization of the synthesized materials

Fig. 1 shows the powder X-ray diffraction patterns of obtained UiO-66-SO$_3$H and MIL-101-SO$_3$H materials. Both X-ray diffractograms showed high crystallinity being in fair agreement with the simulated diffraction patterns of UiO-66 and MIL-101 phases. Nitrogen adsorption-desorption isotherms at 77 K for UiO-66-SO$_3$H and MIL-101-SO$_3$H samples are depicted in Fig. 2a. Both materials showed a significant nitrogen uptake at lower P/P$_o$ values (<0.1), which indicates the presence of micropores. Their textural properties -including reference SAC-13 and Amberlyst-15 catalysts- are summarized in Table 1. It was evidenced a higher surface area and total pore volume for MIL-101-SO$_3$H material, as reported in the literature [11,12,27]. Regarding their pore size distribution (Fig. 2b), UiO-66-SO$_3$H has most of the pores with a diameter centred near 9.5 Å and a small amount centred at 16 Å, while MIL-101-SO$_3$H exhibits a multimodal pore size distribution (micropores and mesopores), with three maxima at 11, 18 and 23 Å. In addition, the presence of the sulfonic groups in the synthesized materials was confirmed by the sulfur content measured through elemental analysis, showing values of 0.68 and 0.91 mmol S/g for MIL-101-SO$_3$H and UiO-66-SO$_3$H, respectively.

3.2. Evaluation of catalytic activity and stability

Acid-based UiO-66-SO$_3$H and MIL-101-SO$_3$H materials were tested as heterogeneous acid catalysts in the acylation of anisole using acetic anhydride as the acylating agent. For comparison purposes, two commercial acid catalysts were also tested: SAC-13 (perfluorosulfonic acid resin Nafion support over macroporous amorphous silica) and Amberlyst-15 (macro reticular polystyrene-based ion exchange resin with strongly acidic sulfonic groups). Textural properties and sulfur concentration of all these materials are shown in Table 1. The catalytic activity was assessed at selected reaction conditions according to our previous work [44]: temperature 423 K, equimolar anisole/acetic anhydride molar ratio, 1.25 wt% of catalyst relative to anisole mass and absence of solvent. Table 2 shows the catalytic performance of all materials in terms of the overall anisole conversion and specific activity considering the content of sufonic-acid sites at 1 h and 5 h of reaction time. As shown, UiO-66-SO$_3$H displays a low anisole conversion even after 5 h of reaction time. This fact is attributed to the limited availability of the acid active sites within its microporous structure. It is

well-known that textural properties and the internal structure of the heterogeneous catalysts are key factors for the catalytic reaction [4]. The UiO-66-SO$_3$H material showed a pore size distribution centred at 9.5 Å which is probably hindered the diffusion of reactants through the porous system to reach the acid sites. For this reason, this material was dismissed for further experiments. On the other hand, MIL-101-SO$_3$H material achieved values of anisole conversion of 9.7% and 14.8%, for 1 h and 5 h, respectively, as well as a high selectivity (over 92%) towards the p-MAP product. The catalytic activity of MIL-101-SO$_3$H can be due to sulfonic Brønsted acid sites but also unsaturated chromium metal centres as Lewis acid sites. It is well known that both type of acid sites can catalyze acylation reactions [45]. Measurements of CO adsorption using ultrahigh vacuum IR spectroscopy evidenced characteristic signals of Brønsted and Lewis acid sites at 2160 cm^{-1} and 2200 cm^{-1}, respectively (see Fig. S1). In order to assess the potential catalytic activity of unsaturated chromium acid centres, the same catalytic test was performed using the MIL-101 material prepared with a non-sulfonated terephthalic acid as linker [46]. A dramatically decrease of activity was observed for the MIL-101 material, achieving hardly 0.9% of anisole conversion after 5 h of reaction (Table 2). Thus, this result evidenced that sulfonic acid groups, as Brønsted acid sites, are responsible for the acylation of anisole.

The catalytic activity of MIL-101-SO$_3$H in terms of the anisole conversion was similar to commercial material SAC-13, characterized as a perfluorosulfonic acid-based catalyst [44]. In contrast, the commercial Amberlyst-15 material exhibited a higher anisole conversion (28% after 5 h of reaction). This fact is mainly attributed to the higher content of sulfonic-acid sites (4.8 mmol S/g) compared to SAC-13 (0.13 mmol S/g) or MIL-101-SO$_3$H (0.68 mmol S/g) as well as the lower diffusion constraints in its open macroporous structure. However, it must be noted the recommended maximum operating temperature for this polymeric resin is 393 K and the reaction temperature used in this case for the acylation reaction was 423 K, so the stability of Amberlyst-15 catalyst was not completely guaranteed. Regarding the selectivity to the p-MAP product, both commercial catalysts also showed selectivity values higher than 92% as the MOFs materials. On the other hand, specific activities in terms of mmol of converted anisole per mmol of S were calculated (Table 2) in order to compare the catalytic performance considering the different content of sulfonic acid groups. In this case, the catalysts with higher specific activities were SAC-13 (399 and 612 mmol anisole/mmol S for 1 h and 5 h of reaction time, respectively) and MIL-101-SO$_3$H (82 and 113 mmol anisole/mmol S for 1 h and 5 h of reaction time, respectively). Amberlyst-15, although providing the highest conversion of anisole, had the lowest activity per acid centre. These results evidenced a relevant anisole conversion of MIL-101-SO$_3$H, although with a lower specific activity per mmol of acid centres as compared to the SAC-13 material. This fact is attributed to the different nature of the acid centres. The acid strength of perfluorosulfonic acid groups in the commercial SAC-13 material is supposed to be superior to that of other sulfonated materials such as the MIL-101-SO$_3$H catalyst due to the presence of the perfluorocarbon chain, in particular to the fluorine atoms in α-position to the sulfonic centres [47]. There are previous works based on studies of ammonia adsorption calorimetry that corroborate this statement according to the higher ammonia absorbing up to saturation coverage (ΔH_{ads} (NH$_3$)) of perfluorosulfonic acid groups (values between −158 and −162 kJ/mol) as compared to sulfonic acid groups of sulfonated materials (values between −110 and −116 kJ/mol) [48,49].

It must be also pointed out that the catalysts showed a moderate increase of the anisole conversion for the longer reaction times (5 h) as compared to the remarkable values achieved at the early reaction time of 1 h. This behaviour is quite usual, as a result of partial deactivation of heterogeneous catalysts in Friedel-Crafts acylation reactions due to the formation of poly-acetylated compounds that are adsorbed over the acid centres [50]. Interestingly, this fact seems to be more accentuated in SAC-13 catalyst and it is likely associated to the higher acid strength of
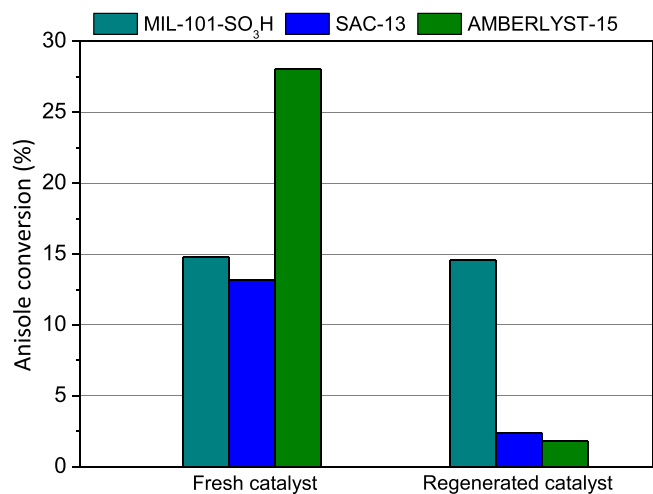
Fig. 3. Catalytic performance of fresh and regenerated catalysts at 5 h.

perfluorosulfonic acid sites. In contrast, the sulfonated Amberlyst-15 resin showed a less significant deactivation, probably due to the high concentration of active centres and hence high ratio of active centres to the acylating agent.

Concerning the stability of the MIL-101-SO$_3$H material in the acid-catalyzed reaction of anisole, X-ray diffraction analysis of the material after the reaction (shown in SI, Fig. S2), did not evidence variations of the characteristic diffraction peaks of the pristine phase. Additionally, the sulfur content in the material remained constant after the reaction, discarding the leaching of sulfonic acid to the reaction medium (results also shown in SI, Table S2) and therefore the potential homogeneous contribution to the catalytic activity.

3.3. Regeneration and reuse of catalysts

Thermogravimetric analysis of the recovered catalysts after reaction (results shown in SI, Fig. S3) evidenced the presence of organic compounds (reagents and poly-acetylated products) adsorbed on the catalyst surface. Previous works of evaluation of heterogeneous catalysts for Friedel-Crafts acylation have proven that spent catalysts can be regenerated by calcination at high temperature or by extraction with boiling HNO$_3$ solution [51,52]. These regeneration treatments are not sustainable from an environmental and economic point of view. In this sense, the use of MOFs as acid catalysts in this reaction allows the use of more sustainable regeneration treatments in order to maintain its catalytic activity. In this work, a new alternative has been explored based on the regeneration of the catalysts by washing with acetone and ethanol to remove weakly adsorbed compounds and further thermal activation at

373 K for 1 h under vacuum to remove stronger coordinated molecules. The results of the anisole conversion for the initial and regenerated catalysts after a first catalytic run are shown in Fig. 3. Interestingly, the catalytic activity of MIL-101-SO$_3$H material was remained constant after regeneration, while both commercial SAC-13 and Amberlyst-15 catalysts could not recover the initial activity with a significant decrease of activity. Thus, for the commercial materials, the regeneration procedure carried out is not effective to eliminate potential poly-acetylated by-products generated during the first use.

Additionally, four successive catalytic runs were carried out for the MIL-101-SO$_3$H material (Fig. 4a). using the same reaction conditions previously tested (423 K, equimolar anisole/acetic anhydride molar ratio, 1.25 wt% of catalyst relative to anisole mass). As it can be seen in Fig. 4a, there was no significant loss of activity along with the four cycles of 5 h of reaction, maintaining an anisole conversion near 15%. To assess the MOF structure stability, X-ray diffraction patterns of the recovered catalyst after some cycles were evaluated (second and fourth ones, Fig. 4b). These results evidenced that the structure remains stable after regeneration in successive catalytic runs. Moreover, the recovery of the solid catalyst was practically complete after each cycle. Additionally, no sulfur was detected in the liquid reaction medium confirming the stability of the catalyst and non-traces of sulfonated linker that could activate homogeneous catalytic reactions. All these results proved that MIL-101-SO$_3$H material is catalytically active, easy to recover and regenerate, and effective for successive reaction cycles in this catalytic system. Therefore, MIL-101-SO$_3$H material is considered a potential attractive acid heterogeneous catalyst to be tested in other acid-catalyzed reactions.b).

4. Conclusions

MIL-101-SO$_3$H and UiO-66-SO$_3$H materials have been evaluated as acid heterogeneous catalysts for Friedel–Craft acylation of anisole with acetic anhydride. The catalytic activity of MIL-101-SO$_3$H proves the presence of available active sulfonic-acid sites with a remarkable specific activity in terms of anisole conversion. The contribution of Lewis acid sites from unsaturated chromiun centres in the acylation of anisole at the studied reaction conditions can be discarded, according to the low activity of the non-sulfonated MIL-101 material. In contrast, UiO-66-SO$_3$H showed a poor anisole conversion due to a lower specific surface area and a porous framework that seems to hinder the accessibility of reagents to its acid active sites. The MIL-101-SO$_3$H material has achieved an overall performance in terms of anisole conversion slightly higher than those obtained for reference commercial perflurosufonic acid-based catalyst, SAC-13. In terms of selectivity towards the p-MAP product, MIL-101-SO$_3$H material and commercial reference catalysts (perfluorosufonic SAC-13 and sulfonated Amberlyst-15 resin) showed values higher than 92%. Moreover, the MIL-101-SO$_3$H material was successfully regenerated by a sustainable method for removing reagents
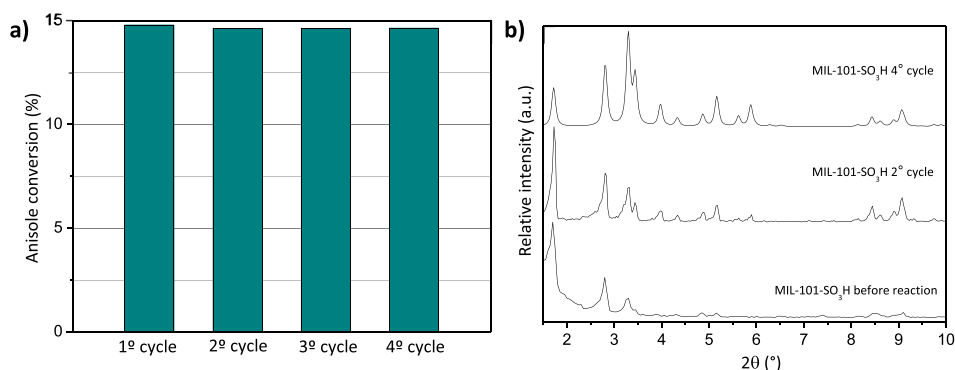

Fig. 4. a) Catalytic activity of MIL-101-SO$_3$H in consecutive reaction cycles of acylation of anisole. b) X-ray diffraction patterns of fresh and used MIL-101-SO$_3$H catalyst after consecutive reactions.

and poly-acetylated products, maintaining its catalytic activity and crystalline structure after 4 successive reaction cycles of 5 h. These results make MIL-101-SO$_3$H a promising greener and effective material for acid-catalyzed chemical reactions.

CRediT authorship contribution statement

Pedro Leo: Conceptualization, Methodology, Investigation, Formal analysis, Writing – original draft. **Neus Crespí**: Conceptualization, Methodology, Investigation, Formal analysis, Writing – original draft. **Carlos Palomino**: Conceptualization, Methodology, Investigation, Formal analysis, Writing – original draft. **Antonio Martin**: Conceptualization, Methodology, Investigation Supervision, Writing –original draft, Writing – review & editing. **Gisela Orcajo**: Conceptualization, Methodology, Investigation Supervision, Writing – review & editing. **Guillermo Calleja**: Supervision, Writing – review & editing, Funding acquisition. **Fernando Martínez** Conceptualization, Methodology, Formal analysis, Resources, Writing – original draft, Writing – review & editing, Funding acquisition.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.cattod.2021.10.007.

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