Full Paper

Comparative Study of the Regioselective Synthesis of β-Aminoalcohols under Solventless Conditions Catalyzed by Sulfated Zirconia and SZ/MCM-41

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Abstract: Sulfated zirconia and SZ/MCM-41 were used as catalysts for the synthesis of β-aminoalcohols via epoxide aminolysis. Sulfated zirconia was prepared by sol-gel and SZ/MCM-41 was obtained by impregnation. Solid catalysts were characterized by XRD, SEM-EDS, UV-Vis, FT-IR pyridine desorption and Nitrogen physisorption. Both acid materials were useful as catalysts, even when they were recycled several times. The β-aminoalcohols were characterized by FT-IR, ^{1}H- and ^{13}C-NMR and GC-MS.

Keywords: Sulfated zirconia, SZ/MCM-41, β-aminoalcohols, oxirane, solventless reactions.

Introduction

Since the discovery that sulfated zirconia (SZ) has a high catalytic activity in organic reactions under mild conditions, it has been the subject of numerous studies on its synthesis, characterization and applications [1]. The strength, nature and structure of sulfated zirconia have been studied for a long time, starting with Arata and Hino [2], who reported that zirconia, upon proper treatment with

sulfuric acid or ammonium sulfate, exhibits 10^4 times stronger acidity than that of 100% sulfuric acid. It is known that sulfated zirconia belongs to the solid superacids family and that the stability, structure and acidity type are directly related to the preparation and sulfation processes. [3] Some authors mention that the strong acidity of sulfated zirconia is due to a combination of Lewis and Brønsted acid sites. Sulfated species with covalent bonds as S=O on the surface of SZ are needed for acidity. Other authors have suggested that water absorption in SZ generates both types of acid sites. In this context, some acid models have been proposed to explain the acid features of SZ [4]. Sulfated zirconia acidity has proven to be useful in organic transformations such as *n*-butane isomerization, [5] Friedel-Craft acylations, [6,7] fine chemical organic transformations, [8] alcohol dehydrations, [9] and polymerizations [10].

Typically, the surface area of solid catalysts is an important factor that directly influences their catalytic performance. Sulfated zirconia exhibits an area of close to 100 m^2g^{-1}. New reports have shown that sulfated zirconia supported on MCM-41 shows an increased surface area, generating a good catalyst for cumene cracking, [11] isomerization of *n*-butane [12] or *n*-pentane, [13] and veratrole acylation [14].

β-Aminoalcohols are present in several biologically active natural and synthetic products; unnatural amino acids, chiral auxiliaries, as chiral catalysts in asymmetric synthesis [15], and their existence is fundamental because of their biological and chemical reactivity. One of the most common synthesis methods for β-aminoalcohols is the opening of oxiranes with heteronucleophiles with nitrogen or oxygen. The classical approach for the synthesis of β-aminoalcohols, involving nucleophilic opening of epoxides by amines requires heat treatment and requires an excess of amine. Several catalysts have been utilized in these reactions: montmorillonite [16], Ti(o-*i*-Pr)$_4$ [17], SmI$_2$, [18] basic metal amides, [19] SmCl$_3$ [20], metal triflates such as Sn(OTf)$_2$, Cu(OTf)$_2$, LiOTf and Yb(OTf)$_3$ [21], ammonium decatungstocerate (IV) [22], RuCl$_3$ [23], zirconium (IV) chloride [24], cyclodextrins [25] and also CeCl$_3$ [26], silica-gel [27], alumina [28], ionic liquids [29], water [30], LiClO$_4$·3H$_2$O [31], and heteropolyacids, for example H$_3$PW$_{12}$O$_{40}$ [32, 33] and K$_5$CoW$_{12}$O$_{40}$·3H$_2$O [34].

We have successfully used sulfated zirconia for Biginelli condensation reactions, [35] for the Paal Knorr reaction in the synthesis of tetrahydroindolone derivatives [36] and for the acylal protection and deprotection reactions [37]. In this paper we have synthesized, characterized and evaluated sulfated zirconia and SZ/MCM-41 in the oxirane opening with amines to obtain β-aminoalcohols under mild, solventless conditions.

Results and Discussion

Figure 1 corresponds to the sulfated zirconia (SZ) tetragonal phase, in correspondence with ICSD collection code: 066787, given by reflections in 2θ = 30.18° (relative intensity is 100) as well as peaks at 34.616°, 35.283°, 50.214°, 50.770°, 59.291°, 60.187° and 63.724° [38].
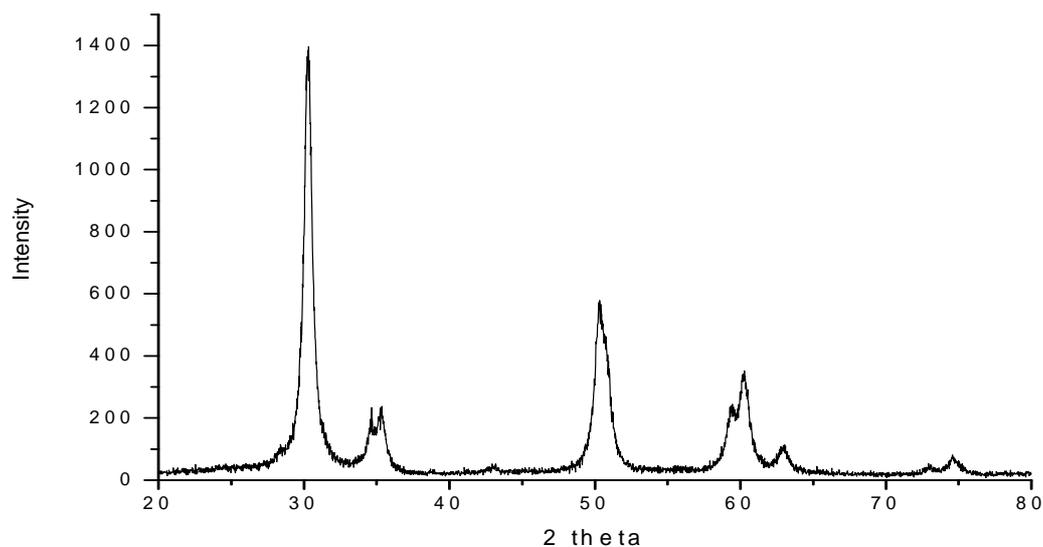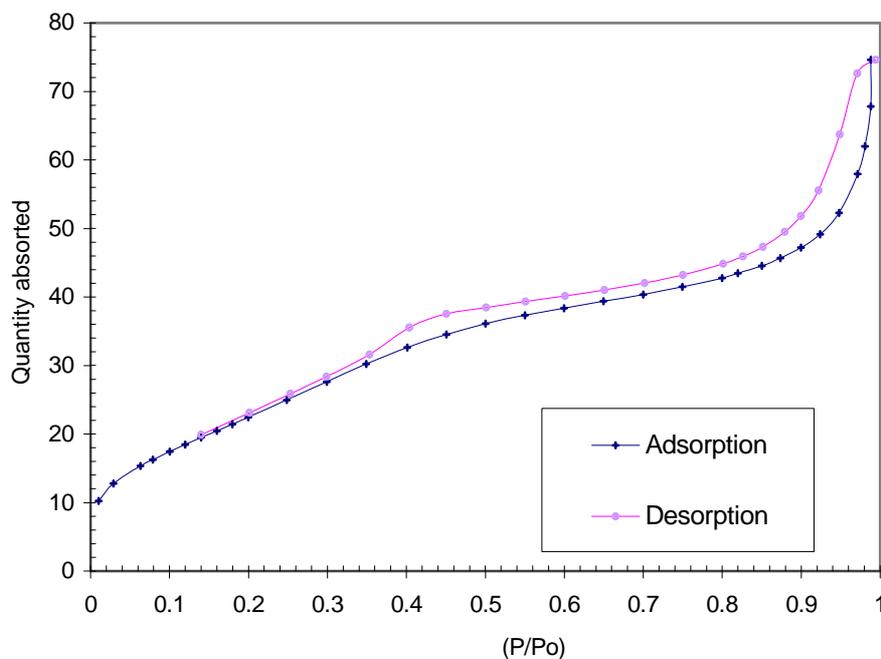
Figure 1. XRD of sulfated zirconia.

Figure 2. Nitrogen adsorption-desorption plot of SZ.

Figure 2 shows the adsorption-desorption plot that gave an isotherm plot type IV in the BET (Brunauer, Emett and Teller) classification; the isotherm hysteresis loop indicated a uniform pore size distribution [39]. The surface area calculated by BET was 86.15 m²g⁻¹, the pore size measured 41.623 Å and the pore volume value was 0.089 cm³g⁻¹. The UV-vis spectrum of sulfated zirconia displays a broad signal centered in 210 nm that is assigned to the tetragonal phase of sulfated zirconia [40]; a similar spectrum is observed for SZ/MCM-41 (Figure 3).
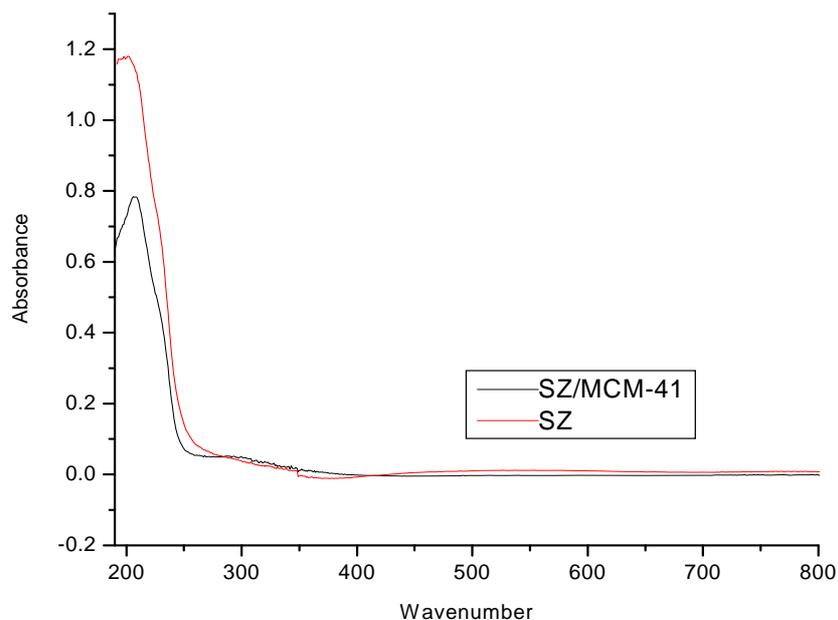
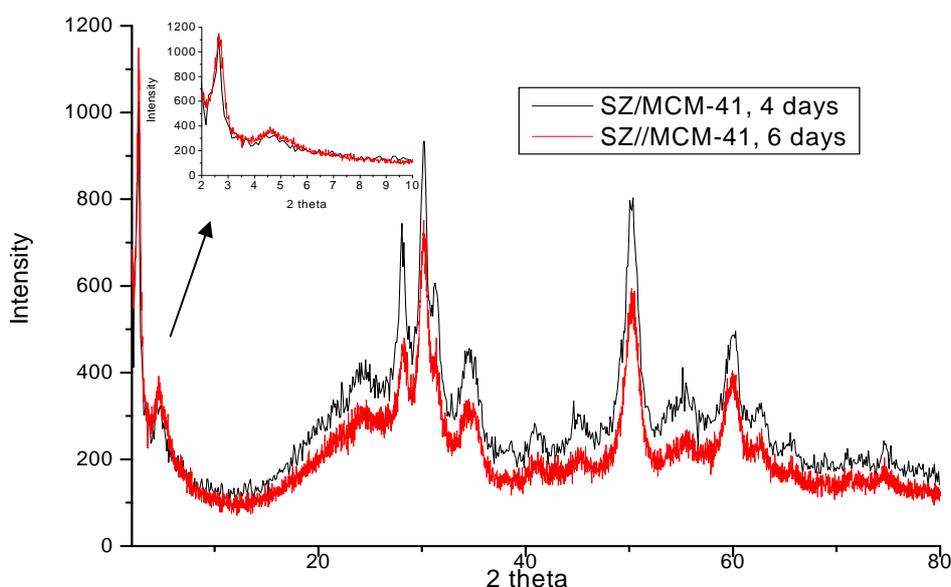**Figure 3.** UV-vis spectra of SZ and SZ/MCM-41.

Figure 4. XRD of SZ/MCM-41 dried 4 and 6 days.

The powder XRD pattern of SZ/MCM-41 shows an intense diffraction peak due to the (100) reflection of MCM-41 at low 2θ ranges, in addition to (110) and (200) in $2\theta = 4.5°$ and $2\theta = 5°$ respectively, which are characteristic of mesoporous materials (Figure 4). When sulfated zirconia is supported in siliceous MCM-41, we observed that the structure of the mesoporous molecular sieve is maintained after loading the SZ, the (100) reflection intensity can be observed, as well as the (110) reflection. We also noted that sulfated zirconia was formed on the support in two phases; monoclinic ($2\theta = 28°$) and tetragonal ($2\theta = 30°, 35°, 50°, 60°$). This is ascribed to the transformation from the metaestable tetragonal phase to the monoclinic phase that is retarded in the presence of promoters. However, an increase of the monoclinic phase was observed when the drying time decreased from 6 to 4 days. The BET surface calculated for SZ/MCM-41 dried for 6 days was 314.92 m^2g^{-1}.

The IR spectra of the sulfated zirconia indicated that pyridine adsorbed in the 1350–1600 cm^{-1} region, as shown in Figure 5, where two strong bands near 1490 and 1450 cm^{-1} and a weak band at 1540 cm^{-1} can be observed when desorption temperature was 50°C. When the temperature was increased, the bands became weaker. The IR pyridine desorption spectra of SZ/MCM-41 showed broad bands at 1490 and 1450 cm^{-1} and a slight and broad band at 1540 cm^{-1}. In general, the band at 1445 cm^{-1} is assigned to the adsorption of pyridine on Lewis acidic sites, and the band at 1545 cm^{-1} is associated to the adsorption on Brønsted acidic sites [11].
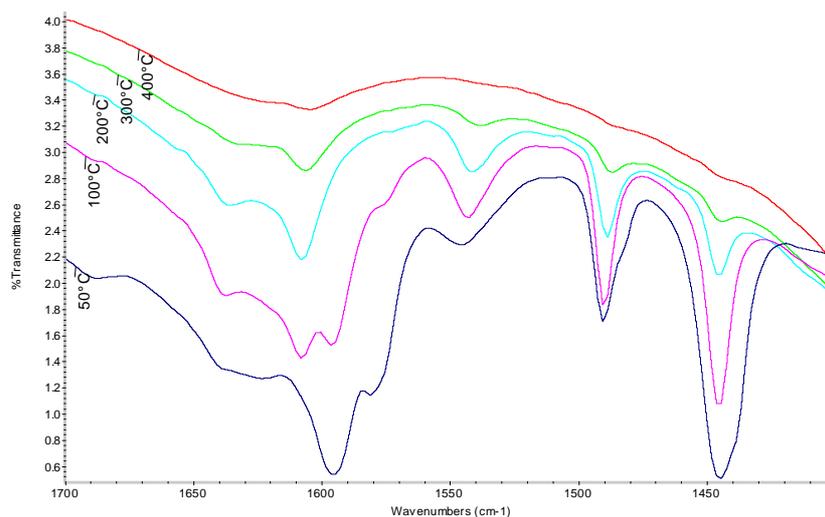

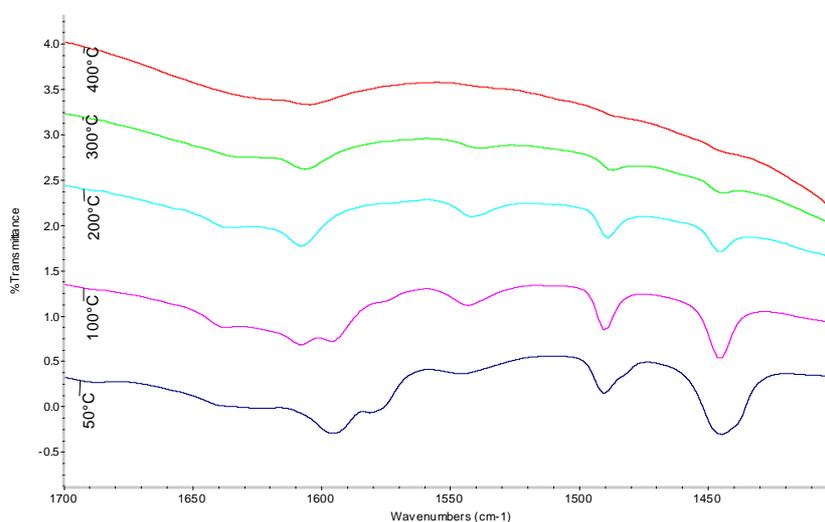**Figure 5.** IR spectra of desorption of pyridine from SZ.

Figure 6. IR spectra of desorption of pyridine from SZ/MCM-41.

These results indicate that the acidic sites over sulfated zirconia and SZ/MCM-41 correspond mainly to Lewis species. The calculated amounts of pyridine adsorbed on both types of acid sites at different desorption temperatures are reported in Table 1.

Table 1. Acidity measurement results for SZ and SZ/MCM-41.

Temperature (°C)	Sulfated Zirconia (µmol py g^{-1})			SZ/MCM-41 (µmol py g^{-1})		
	Brønsted	**Lewis**	**Total**	**Brønsted**	**Lewis**	**Total**
50	101	179	280	18	224	242
100	69	74	143	30	71	101
200	33	52	85	18	17	35
300	14	27	41	5	5	10

The SEM-EDS technique was used to observe and quantify SZ dispersion on MCM-41. Figure 7 shows thin layers of SZ crystallized and dispersed on the support. The semi-quantitative analysis of the sample performed by EDS and the corresponding emission spectra of SZ/MCM-41 are shown in Figure 8.
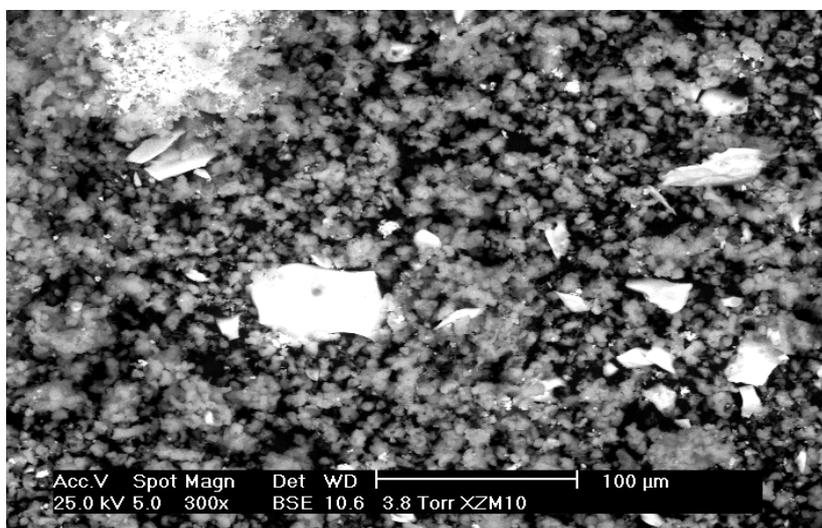
Figure 7. SEM image of SZ/MCM-41.

Element	Wt %
O	40.94
Si	31.38
S	2.32
Zr	25.36
Total	100
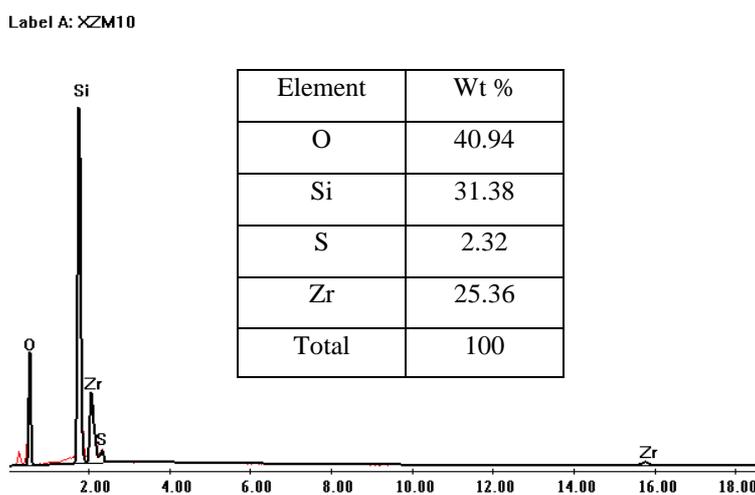
Figure 8. Composition of SZ/MCM-41.

The results of the experiments related to Scheme 1 are presented and compared in the Table 2.

Scheme 1. Aminolysis of oxiranes.
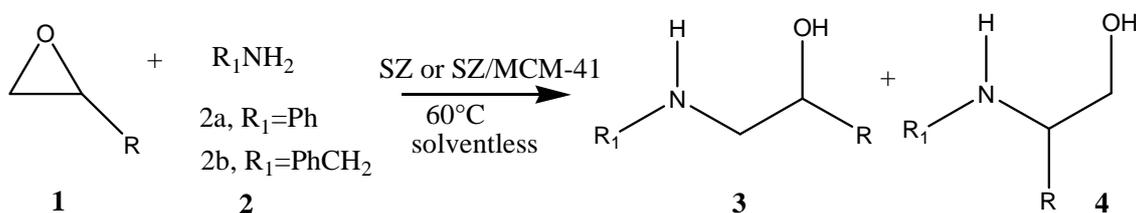
Recycling tests were performed for SZ and SZ/MCM-41. Once the catalysts were used, recovered, and reactivated at 550°C for 1 h, they were utilized in the reaction again. Entry 15 (2, 3-Epoxy-3-phenoxy propane) and benzylamine were employed as reagents. When the recycled catalysts were tested no significant changes in yield were observed, neither with SZ nor with SZ/MCM-41. XRD analysis was performed in order to notice that an increase of the monoclinic phase ($2\theta = 28°$), after the SZ sample was reactivated three times (Figure 9). SZ/MCM-41 is predominantly monoclinic after three recycling periods as shown in figure 10.
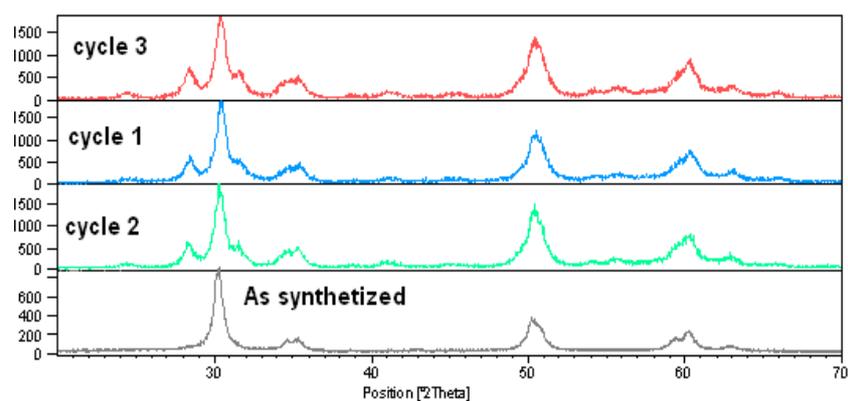

Figure 9. XRD patterns of reactivated SZ.

Position [°2 theta]
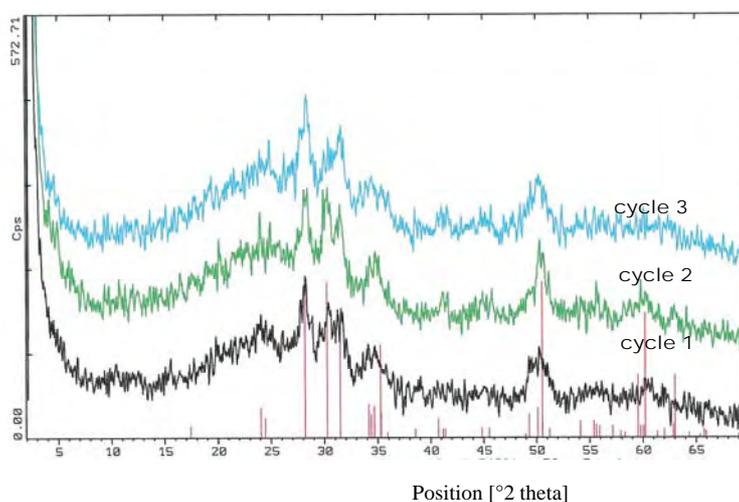
Figure 10. XRD patterns of reactivated SZ/MCM-41.

Table 2. SZ and SZ/MCM-41 as catalysts.

Entry	1	2	Time (h)	3/4 Yield (%) Cat. = SZ	3/4 Yield (%) Cat. = SZ/MCM-41
1		2a	0.5	74/0	23/0
2 [41,42]*		2a	5	61/20	63/0
3 [43]		2b	6	82/0	35/0
4*		2a	5	63/13	55/0
5		2b	5	81/0	54/0
6 [44,45(b,c)]		2a	5	97/0	84/0
7		2b	5	71/0	14/0
8 [45]		2a	5	93/0	99/0
9		2b	5	83/0	45/0
10 [46](d), 45(c), 42]*		2a	1	0/96	5/91
11 [41,47]		2b	6	53/31	55/25
12 [41]		2a	5	81/0	83/0
13		2b	5	87/0	81/0
14 [41, 46]		2a	0.5	86/0	81/0
15 [41]		2b	0.5	87/0	52/0
16		2a	1	76/0	57/0
17		2b	1	77/0	51/0
18		2a	1	76/0	59/0
19 [48]		2b	1	85/0	82/0
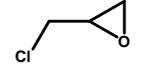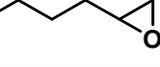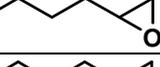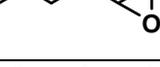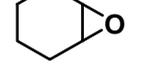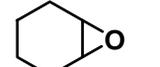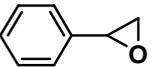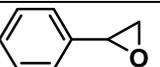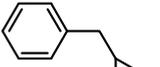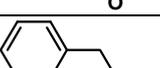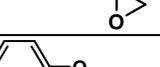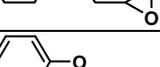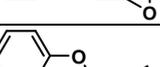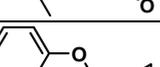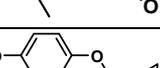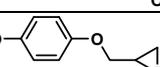
Yields of the isolated possible products **3** or **4** shown in Scheme 1; the regioselectivity was evaluated by GC-MS; * the structures of the minor products in entries 2, 4 and 10 were determined by CI mode GC-MS.

Sulfated zirconia and SZ/MCM-41 were used as catalysts in the β-aminoalcohols synthesis in agreement with scheme 1. The specific area increase of the SZ/MCM-41 compared to that of the SZ does not reflect in a better yield in the epoxide aminolysis reactions, which may be attributed to an overall greater acid strength of the former; at the same time a better regioselectivity was noticed in entries 2 and 4. Thus, the surface properties seem to play a secondary role in catalytic activity in comparison to other factors, such as acidity [49].

Conclusions

In summary, we have described a simple procedure for the synthesis of β-aminoalcohols using SZ and SZ/MCM-41. The catalysts were recovered and reused without any appreciable loss of their activity.

Experimental

General

Powder X-ray diffraction (XRD) patterns were obtained with an X´Pert instrument using Cu Kα radiation. Nitrogen adsorption/desorption isotherms were plotted at -196 °C on a Micromeritics ASAP 2020 equipment. IR spectra were recorded on a Perkin-Elmer FT-IR system/GX spectrometer. SEM-EDS analysis was performed using a SEM XL30 ESEM microscopy. The reaction products were analyzed by means of a Hewlett Packard 6890/5973 GC/MS system equipped with an HP-5 column, with the oven program 70-200°C (10°C/min) for 4 min then, 200-280°C (10°C/min) for 3 min, inj. 250 °C, det. 280 °C; the detector was set in the Chemical Ionization mode using methane as reactive gas. ^{1}H-NMR and ^{13}C-NMR spectra were measured at 500 and 125 MHz, respectively, on a Bruker Avance DMX-500 spectrometer using CDCl$_3$ as solvent and tetramethylsilane as internal standard. Pyridine adsorption infrared spectra test was carried out in a Nicolet 750 spectrometer.

Sulfated zirconia synthesis

Zirconium *n*-propoxide (20 mL, 70 % n-propanol) were mixed with 2-propanol (30 mL) and stirred with a magnetic bar. Then, distilled water (3.2 mL) was mixed with sulfuric acid (1 mL, 98%) and this solution was added dropwise in order to carry out the hydrolysis and gelation of the zirconium *n*-propoxide. The solid was dried at 80°C until complete alcohol evaporation and then calcined in air at 600°C for 6 h.

MCM-41 synthesis

Pure siliceous MCM-41 was prepared from a gel described elsewhere [50]. Mixing cetyltrimetylammonium bromide (7.289 g) with deionized water (200 mL) at 30°C gave a clear solution, and then triethylamine (3.34 mL) was added. After stirring for a few minutes, TMAOH (18 mL, 10 wt %) and finally TEOS (22.35 mL) were added. The resulting gel, with molar composition 1 TEOS : 0.2 CTMABr : 0.6 EA : 0.2 TMAOH : 150 H$_2$O, was placed in glass bottles and sonicated for

4 h. The precipitated solid was recovered by filtration and washed with deionized water, dried at 60 °C overnight and calcinated at 540 °C for 6 h under air flow.

Synthesis of sulfated zirconia supported on MCM-41

Preparation of the sulfated zirconia supported in MCM-41 (SZ/MCM-41) has been already described [13]. In a first procedure, siliceous MCM-41 (300 g) were mixed with zirconium sulfate (15 mL) in methanol (1 wt %), the heterogeneous mixture was stirred 14 h. Then, the impregnated solid was dried for 6 days at 50 °C and finally calcinaed at 660 °C for 3 h in air flow.

β-Aminoalcohol synthesis

In a general procedure oxirane **1** (1 mmol) and nucleophile (aniline or benzylamine, **2**, 1.1 mmol) were placed in a vial equipped with a magnetic stirrer and plastic cap. The mixture was heated at 60 °C in the presence of sulfated zirconia or sulfated zirconia on MCM-41 (50 mg) without solvent (Scheme 1). After the reaction, the mixture was left at room temperature, the catalyst was recovered by filtration and the organic product was dried in a rotavapor under reduced pressure and the products were purified by silica gel or alumina column chromatography. All products were identified by FR-IR, ^{1}H-NMR, ^{13}C-NMR and by comparison with the previously described physical and spectroscopic data of the β-aminoalcohols.

1-Chloro-3-(phenylamino)propan-2-ol (Entry 1). GC-MSD for $C_9H_{12}NOCl$ (mw: 185.66 g/mol): $[M+1]^+ = 186$, $[M+29]^+ = 214$, $[M+41]^+ = 226$, $[M-17]^+ = 168$; IR (cm^{-1}): 3396, 1602, 1499, 1419, 1314, 1261, 1161, 1156, 1088, 1075, 958, 851, 823, 748, 692; ^{1}H-NMR: δ (ppm) 7.16-7.21 (m, 2H, Ar-H), 6.75 (tt, 1H, $J_1= 1.0$, $J_2= 7.3$ Hz, Ar-H), 6.65 (dd, 2H, $J_1= 1.0$, $J_2= 8.6$ Hz, Ar-H), 4.01-4.06 (m, 1H, -CH-OH), 3.63 (dd, 1H, $J_1= 4.5$, $J_2= 11.3$ Hz, -CH-Cl), 3.58 (dd, 1H, $J_1= 6.0$, $J_2= 11.3$ Hz , -CH-Cl), 3.46 (bs, 2H, -OH, -NH), 3.33 (dd, 1H, $J_1= 4.3$, $J_2= 13.3$ Hz, -NH-CH-CH-OH), 3.17 (dd, 1H, $J_1= 7.4$, $J_2= 13.3$ Hz, -NH-CH-CH-OH); ^{13}C-NMR: δ (ppm) 147.42, 129.32, 118.36, 113.39, 69.65, 47.52, 47.15.

1-(Phenylamino)hexan-2-ol (Entry 2). m.p.: 51-52 °C; GC-MSD for $C_{12}H_{12}NO$ (mw: 193.29 g/mol): $[M+1]^+ = 194$, $[M+29]^+ = 222$, $[M+41]^+ = 234$, $[M-17]^+ = 176$, IR (cm^{-1}): 3273, 2969, 2929, 2831, 1602, 1499, 1460, 1436, 1380, 1309, 1251, 1222, 1139, 1084, 1009, 984, 886, 844; ^{1}H-NMR: δ (ppm) 7.16-7.20 (m, 2H, Ar-H), 6.72 (tt, 1H, $J_1= 1.0$, $J_2= 7.3$ Hz, Ar-H), 6.63-6.66 (m, 2H, Ar-H), 3.94 (bs, 1H, -OH or NH), 3.80-3.85 (m, 1H, -CH-OH), 3.26 (dd, 1H, $J_1= 3.2$, $J_2= 12.8$ Hz -NH-CH-CH-OH), 2.99 (dd, 1H, $J_1= 8.6$, $J_2= 12.8$ Hz -NH-CH-CH-OH), 1.98 (bs, 1H, -OH or NH), 1.49-1.56 (m, 2H, -CH$_2$-CH-OH), 1.31-1.48 (m, 4H, - CH$_2$-CH$_2$-CH$_3$), 0.92 (t, 3H, $J= 7.3$ Hz, -CH$_2$-CH$_3$); ^{13}C-NMR: δ (ppm) 148.30, 129.29, 117.87, 113.26, 70.41, 50.33, 34.82, 27.80, 22.74, 14.04.

1-(Benzylamino)hexan-2-ol (Entry 3). GC-MSD for $C_{13}H_{21}NO$ (mw: 207.32 g/mol): $[M+1]^+ = 208$, $[M+29]^+ = 236$ $[M+41]^+ = 248$, $[M-17]^+ = 190$; IR (cm^{-1}): 2927, 1635, 1453, 1026, 735, 696; ^{1}H-NMR: δ (ppm) 7.24-7.27 (m, 2H, Ar-H), 7.30-7.35 (m, 3H, Ar-H), 3.83 (d, 1H, $J= 13.2$ Hz, -NH-CH-Ar), 3.78 (d, 1H, $J= 13.2$ Hz -NH-CH-Ar), 3.60-3.67 (m, 1H, -CH-OH), 2.75 (dd, 1H, $J_1= 3.0$, $J_2= 12.0$ Hz

-NH-C<u>H</u>-CH-OH), 2.47 (dd, 1H, J_1= 9.4, J_2= 12.0 Hz -NH-C<u>H</u>-CH-OH), 2.36 (bs, 2H, -O<u>H</u>, N<u>H</u>), 1.25-1.46 (m, 6H, -C<u>H</u>$_2$-CH$_3$), 0.89 (t, 3H, *J*= 7.2 Hz, -CH$_2$-C<u>H</u>$_3$); ^{13}C-NMR: δ (ppm) 139.79, 128.49, 128.13, 69.53, 54.72, 53.59, 34.73, 27.80, 22.77, 14.01.

1-(Phenylamino)hex-5-en-2-ol (Entry 4). m.p.: 32-33 °C; GC-MSD for C$_{12}$H$_{17}$NO (mw: 191.27 g/mol): [M+1]$^+$ = 192, [M+29]$^+$ = 220, [M+41]$^+$ = 232. IR (cm^{-1}): 3282, 2936, 2933, 2912, 2833, 1640, 1603, 1496, 1463, 1434, 1414, 1300, 1250, 1221, 1182, 1108, 1072, 1022, 997, 912; ^{1}H-NMR: δ (ppm) 7.15-7.20 (m, 2H, Ar-<u>H</u>), 6.73 (tt, 1H, , J_1= 1.0, J_2= 7.3 Hz, Ar-<u>H</u>), 6.63-6.66 (m, 2H, Ar-<u>H</u>), 5.81-5.89 (m, 1H, -C<u>H</u>=CH$_2$), 5.07 (ddd, 1H, J_1= 1.6, J_2= 3.5, J_3= 17.1 Hz, -CH$_2$-CH=C<u>H</u>), 4.98-5.01 (m, 1H, -CH$_2$-CH=C<u>H</u>), 3.98 (bs, 1H, -O<u>H</u> or N<u>H</u>), 3.83-3.88 (m, 1H, -C<u>H</u>-OH), 3.26 (dd, 1H, J_1= 3.3, J_2= 12.9 Hz, -NH-C<u>H</u>-CH-OH), 3.01 (dd, 1H, J_1= 8.5, J_2= 12.9 Hz, -NH-C<u>H</u>-CH-OH), 2.14-2.30 (m, 2H, -C<u>H</u>$_2$-CH=CH$_2$), 2.10 (bs, 1H, -O<u>H</u> or -N<u>H</u>), 1.63 (ddd, 2H, J_1= 6.4, J_2= 7.7, J_3= 7.7 Hz, -CH$_2$-C<u>H</u>$_2$-CH-OH); ^{13}C-NMR: δ (ppm) 148.20, 138.12, 129.27, 117.92, 115.14, 113.28, 69.82, 50.22, 34.05, 29.92.

1-(Benzylamino)hex-5-en-2-ol (Entry 5). GC-MSD for C$_{13}$H$_{19}$NO (mw: 205.3 g/mol): [M+1]$^+$ = 206, [M+29]$^+$ = 234, [M+41]$^+$ = 246, [M-17]$^+$ = 188. IR (cm^{-1}): 2918, 2836, 1700, 1638, 1444, 1134, 1043, 988, 906, 852, 727, 693; ^{1}H-NMR: δ (ppm) 7.29-7.35 (m, 4H, Ar-<u>H</u>), 7.25-7.28 (m, 1H, Ar-<u>H</u>), 5.78-5.86 (m, 1H, -C<u>H</u>=CH$_2$), 4.94-5.05 (m, 2H, -CH=C<u>H</u>$_2$), 3.83 (d, 1H, *J*= 13.2 Hz, -NH-C<u>H</u>-Ar), 3.77 (d, 1H, *J*= 13.2 Hz -NH-C<u>H</u>-Ar), 3.63-3.68 (m, 1H, -C<u>H</u>-OH), 2.75 (dd, 1H, J_1= 3.0, J_2= 12.0 Hz, -NH-C<u>H</u>-CH-OH), 2.48 (dd, 1H, J_1= 9.4, J_2= 12.0 Hz, -NH-C<u>H</u>-CH-OH), 2.38 (bs, 2H, -O<u>H</u>, -N<u>H</u>), 2.06-2.26 (m, 2H, -C<u>H</u>$_2$-CH=CH$_2$), 1.43-1.57 (m, 2H, -CH$_2$-C<u>H</u>$_2$-CH-OH); ^{13}C-NMR: δ (ppm) 139.73, 138.39, 128.47, 128.11, 127.16, 114.74, 68.89, 54.55, 53.54, 34.07, 29.92.

2-(Phenylamino)cyclopentanol (Entry 6). m.p.: 56-57 °C; GC-MSD for C$_{11}$H$_{15}$NO (mw: 177.25 g/mol): [M+1]$^+$ = 178, [M+29]$^+$ = 206, [M+41]$^+$ = 218, [M-17]$^+$ = 160; IR (cm^{-1}): 3397, 2957, 1599, 1506, 1482, 1436, 1316, 1259, 1182, 1154, 1112, 1033, 973, 752, 694; ^{1}H-NMR: δ (ppm) 7.20-7.24 (m, 2H, Ar-<u>H</u>), 6.69-6.77 (m, 3H, Ar-<u>H</u>), 4.06-4.09 (m, 1H, -C<u>H</u>-OH), 3.62-3.65 (m, 1H, NH-C<u>H</u>), 3.23 (bs, 2H, -O<u>H</u>, -N<u>H</u>), 2.27-2.33 (m, 1H, C<u>H</u>$_2$-CH-OH), 2.01 (ddd, 1H, J_1= 1.9, J_2= 6.5, J_3= 13.1 Hz, C<u>H</u>$_2$-CH-OH), 1.73-1.90 (m, 2H, C<u>H</u>$_2$-CH-NH), 1.63-1.70 (m, 1H, CH$_2$-C<u>H</u>), 1.39-1.46 (m, 1H, CH$_2$-C<u>H</u>); ^{13}C-NMR: δ (ppm) 147.71, 129.24, 117.49, 113.31, 78.18, 62.06, 32.82, 31.15, 20.98.

2-(Benzylamino)cyclopentanol (Entry 7). m.p.: 42-42 °C; GC-MSD for C$_{12}$H$_{17}$NO (mw: 191.28 g/mol): [M+1]$^+$ = 192, [M+29]$^+$ = 220, [M+41]$^+$ = 232, [M-17]$^+$ = 174. IR (cm^{-1}): 2941, 1452, 1062, 734, 696; ^{1}H-NMR: δ (ppm) 7.30-7.32 (m, 4H, Ar-<u>H</u>), 7.22-7.27 (m, 1H, Ar-<u>H</u>), 3.89 (dd, 1H, J_1= 6.4, J_2= 12.6 Hz, -C<u>H</u>-OH), 3.83 (d, 1H, *J*= 13.0 Hz, -NH-C<u>H</u>-Ar), 3.74 (d, 1H, *J*= 13.0 Hz, -NH-C<u>H</u>-Ar), 2.88 (ddd, 1H, J_1= 5.8, J_2= 7.4, J_3= 7.5 Hz, HO-CH-C<u>H</u>-NH-), 2.34 (bs, 2H, -O<u>H</u>, -N<u>H</u>), 1.92-2.05 (m, 2H, C<u>H</u>$_2$-CH-OH), 1.61-1.74 (m, 2H, C<u>H</u>$_2$-CH-NH), 1.48-1.55 (m, 1H, CH$_2$-C<u>H</u>), 1.30-1.38 (m, 1H, CH$_2$-C<u>H</u>); ^{13}C-NMR: δ (ppm) 139.94, 128.25, 128.25, 128.46, 127.09, 66.29, 52.53, 32.63, 30.12, 20.39.

2-(Phenylamino)cyclohexanol (Entry 8). m.p.: 52-53 °C; GC-MSD for C$_{12}$HN$_{17}$O (mw: 191.28 g/mol): [M+1]$^+$ = 192, [M+29]$^+$ = 220, [M+41]$^+$ = 232, [M-17]$^+$ = 174; IR (cm^{-1}): 3395, 2939, 2923, 2856, 1599, 1513, 1497, 1450, 1436, 1321, 1306, 1259, 1241, 1182, 1152, 1101, 1055, 1048, 1037, 992, 932,

864, 849, 740, 688; ^{1}H-NMR: δ (ppm) 7.17 (t, 2H, *J*= 7.4 Hz, Ar-H̱), 6.73 (t, 1H, *J*= 7.3 Hz, Ar-H̱), 6.69 (dd, 2H, *J₁*= 0.9, *J₂*= 8.5 Hz, Ar-H̱), 3.32 (ddd, 1H, *J₁*= 4.3, *J₂*= 9.4, *J₃*= 10.2 Hz, -CH̱-OH), 3.11 (ddd, 1H, *J₁*= 4.0, *J₂*= 9.3, *J₃*= 11.3 Hz, -CH̱-NH), 3.07 (bs, 2H, -OH̱, -NH̱), 2.08-2.11 (m, 2H, CH̱₂-CH-OH), 1.65-1.69 (m, 1H, CH̱₂-CH-NH), 1.23-1.41 (m, 3H), 0.97-1.05 (m, 1H); ^{13}C-NMR: δ (ppm) 147.75, 129.23, 118.18, 114.24, 74.35, 33.08, 31.50, 24.90, 24.20.

2-(Benzylamino)cyclohexanol (Entry 9). m.p.: 64-65 °C; GC-MSD for $C_{13}H_{19}NO$ (mw: 205.31 g/mol): $[M+1]^+$ = 206, $[M+29]^+$ = 234, $[M+41]^+$ = 246, $[M-17]^+$ = 188. IR (cm^{-1}): 3294, 3060, 2925, 2852, 1603, 1499, 1449, 1429, 1351, 1339, 1224, 1198, 1153, 1099, 1076, 1060, 1029, 972, 920, 893, 880, 845, 830, 747; ^{1}H-NMR: δ (ppm) 7.28-7.33 (m, 4H, Ar-H̱), 7.21-7.25 (m, 1H, Ar-H̱), 3.93 (d, 1H, *J*= 12.9 Hz, -NH-CH̱-Ar), 3.67 (d, 1H, *J*= 12.9 Hz, -NH-CH̱-Ar), 3.18-3.23 (m, 1H, -CH̱-OH), 2.83 (bs, 2H, -OH̱, -NH̱), 2.30 (ddd, 1H, *J₁*= 2.3, *J₂*= 7.4, *J₃*= 9.3 Hz, HO-CH-CH̱-NH-CH₂-Ar), 2.10-2.15 (m, 1H), 1.93-2.02 (m, 1H), 1.64-1.74 (m, 2H), 1.14-1.29 (m, 3H), 0.96-1.04 (m, 1H); ^{13}C-NMR: δ (ppm) 140.05, 128.36, 128.08, 126.95, 73.45, 62.95, 50.62, 33.37, 30.20, 24.92, 24.25.

2-Phenyl-2-(phenylamino)ethanol (Entry 10). GC-MSD for $C_{12}H_{15}NO$ (mw: 213.28 g/mol): $[M+1]^+$ = 214, $[M+29]^+$ = 242, $[M+41]^+$ = 254, $[M-17]^+$ = $[M-17]^+$ = 196, $[M-31]^+$ = 182; IR (cm^{-1}): 3388, 1600, 1503, 1452, 1429, 1352, 1314, 1266, 1180, 1154, 1065, 1025, 993; ^{1}H-NMR: δ (ppm) 7.31-7.37 (m, 4H, Ar-H̱), 7.26 (tt, 1H, *J₁*= 1.6, *J₂*= 7.1 Hz, Ar-H̱), 7.07-7.11 (m, 2H, Ar-H̱), 6.68 (tt, 1H, , *J₁*= 1.0, *J₂*= 7.3 Hz, Ar-H̱), 6.55-6.58 (m, 2H, Ar-H̱), 4.50 (dd, 1H, *J₁*= 4.2, *J₂*= 7.0 Hz, -CH̱-NH), 4.47 (bs, 1H, -OH̱ or -NH̱), 3.93 (dd, 1H, *J₁*= 4.2, *J₂*= 11.1 Hz, CH-CH̱-OH), 3.75 (dd, 1H, *J₁*= 7.0, *J₂*= 11.1 Hz, CH-CH̱-OH), 1.74 (bs, 1H, -OH̱ or -NH̱); ^{13}C-NMR: δ (ppm) 147.20, 140.09, 129.17, 128.83, 127.61, 126.72, 117.87, 113.84, 67.36, 59.85.

2-(Benzylamino)-2-phenylethanol (Entry 11). m.p.: 97-99 °C; GC-MSD for $C_{15}H_{17}NO$ (mw: 227.31 g/mol): $[M+1]^+$ = 228, $[M+29]^+$ = 256 $[M+41]^+$ = 268, $[M-17]^+$ = 210, $[M-31]^+$ = 196; IR (cm^{-1}): 3058, 3025, 1599, 1494, 1452, 1351, 1196, 1116, 1067, 1047, 1027, 911, 850; ^{1}H-NMR: δ (ppm) 7.35-7.38 (m, 2H, Ar-H̱), 7.27-7.33 (m, 7H, Ar-H̱), 7.23-7.26 (m, 1H, Ar-H̱), 3.81 (dd, 1H, *J₁*= 4.4, *J₂*= 8.7 Hz, Ar-CH̱-NH), 3.76 (d, 1H, *J*= 13.0 Hz, Ar-CH̱-NH), 3.70 (dd, 1H, *J₁*= 4.5, *J₂*= 10.8 Hz, CH-CH̱-OH), 3.60 (d, 1H, *J*= 13.0 Hz, Ar-CH̱-NH), 3.55 (dd, 1H, *J₁*= 8.7, *J₂*= 10.8 Hz, CH-CH̱-OH), 2.15 (bs, 2H, -OH̱ and -NH̱); ^{13}C-NMR: δ (ppm) 140.42, 139.99, 128.70, 128.43, 128.21, 127.69, 127.27, 127.08, 66.74, 63.75, 51.18.

2-(Benzylamino)-1-phenylethanol (Entry 11). GC-MSD for $C_{15}H_{17}NO$ (mw: 227.31 g/mol): $[M+1]^+$ = 228, $[M+29]^+$ = 256 $[M+41]^+$ = 268, $[M-17]^+$ = 210; IR (cm^{-1}): 3062, 2924, 2835, 1600, 1490, 1453, 1433, 1346, 1334, 1207, 1182, 1099, 1064, 1037, 1024, 978, 917, 875, 853; ^{1}H-NMR: δ (ppm) 7.29-7.37 (m, 8H, Ar-H̱), 7.24-7.28 (m, 2H, Ar-H̱), 4.73 (dd, 1H, *J₁*= 3.6, *J₂*= 8.9 Hz, -CH̱-OH), 3.86 (d, 1H, *J*= 13.3 Hz, Ar-CH̱-NH), 3.81 (d, 1H, *J*= 13.3 Hz, Ar-CH̱-NH), 2.94 (dd, 1H, *J₁*= 3.6, *J₂*= 12.2 Hz, CH-CH̱-Ar), 2.75 (dd, 1H, *J₁*= 8.9, *J₂*= 12.2 Hz, CH-CH̱-Ar), 2.24 (bs, 2H, -OH̱ and -NH̱); ^{13}C-NMR: δ (ppm) 142.38, 128.49, 128.37, 128.10, 127.52, 127.18, 125.81, 71.76, 56.49, 53.50.

1-Phenyl-3-(phenylamino)propan-2-ol (Entry 12). m.p.: 63-64 °C; GC-MSD for $C_{15}H_{17}NO$ (mw: 227.31 g/mol): $[M+1]^+$ = 228, $[M+29]^+$ = 256, $[M+41]^+$ = 268, $[M-17]^+$ = 210; IR (cm^{-1}): 3342, 2927,

1600, 1559, 1557, 1494, 1454, 1433, 1411, 1314, 1272, 1249, 1179, 1079, 1063, 1027, 830, 741, 687; ^{1}H-NMR: δ (ppm) 7.30-7.35 (m, 2H, Ar-$\underline{H}$), 7.23-7.28 (m, 4H, -N$\underline{H}$, Ar-$\underline{H}$), 7.15-7.19 (m, 2H, Ar-$\underline{H}$), 6.71-6.75 (m, 1H, Ar-$\underline{H}$), 6.61-6.64 (m, 2H, Ar-$\underline{H}$), 4.00-4.10 (m, 1H, -C$\underline{H}$-OH), 3.30 (dd, 1H, J_1= 3.4, J_2= 12.9 Hz, -NH-C$\underline{H}$-CH-OH), 3.08 (dd, 1H, J_1= 8.1, J_2= 12.9 Hz, -NH-C$\underline{H}$-CH-OH), 2.88 (dd, 1H, J_1= 5.2, J_2= 13.6 Hz, Ar-C$\underline{H}$-CH-OH), 2.82 (dd, 1H, J_1= 7.8, J_2= 13.6 Hz, Ar-C$\underline{H}$-CH-OH), 2.24 (bs, 1H, -O$\underline{H}$ or -N$\underline{H}$), 1.72 (bs, 1H, -O$\underline{H}$ or-N$\underline{H}$); ^{13}C-NMR: δ (ppm) 148.15, 137.74, 129.35, 129.23, 128.65, 126.66, 117.84, 113.30, 71.14, 49.44, 41.63.

1-(Benzylamino)-3-phenylpropan-2-ol (Entry 13). m.p.: 58-61°C; GC-MSD for $C_{16}H_{19}NO$ (mw: 241.34 g/mol): [M+1]$^+$ = 242, [M+29]$^+$ = 270, [M+41]$^+$ = 282, [M-17]$^+$= 224; IR (cm^{-1}): 3149, 3061, 3027, 2842, 1602, 1493, 1452, 1425, 1359, 1342, 1178, 1113, 1103, 1080, 1029, 1014, 896, 861, 792, 748, 697; ^{1}H-NMR: δ (ppm) 7.17-7.33 (m, 10H, Ar-$\underline{H}$), 3.83-3.87 (m, 1H, -C$\underline{H}$-OH), 3.75 (d, 1H, J= 13.2 Hz, -NH-C$\underline{H}$-Ar), 3.68 (d, 1H, J= 13.2 Hz, -NH-C$\underline{H}_2$-Ar), 2.62-2.76 (m, 5H, -NH-C$\underline{H}_2$-CH-OH, -N$\underline{H}$, -O$\underline{H}$, HO-CH-C$\underline{H}$-Ar), 2.52 (dd, 1H, J_1= 9.0, J_2= 12.0 Hz, HO-CH-C$\underline{H}$-Ar); ^{13}C-NMR: δ (ppm) 139.65, 138.31, 129.27, 128.36, 128.11, 127.05, 126.25, 70.55, 54.01, 53.49, 41.58.

1-Phenoxy-3-(phenylamino)propan-2-ol (Entry 14). m.p.: 52-53 °C; GC-MSD for $C_{15}H_{17}NO_2$ (mw: 243.31 g/mol): [M+1]$^+$ = 244, [M+29]$^+$ = 272, [M+41]$^+$ = 284, [M-17]$^+$ = 226. IR (cm^{-1}): 3333, 1600, 1588, 1508, 1496, 1467, 1437, 1303, 1242, 1160, 1080, 1046, 995, 942, 884, 815, 750; ^{1}H-NMR: δ (ppm) 7.28 (dd, 2H, J_1= 7.4, J_2= 8.7 Hz, Ar-$\underline{H}$), 7.17 (dd, 2H, J_1= 7.3, J_2= 8.6 Hz, Ar-$\underline{H}$), 6.97 (t, 1H, J= 7.4 Hz, Ar-$\underline{H}$), 6.90 (dd, 2H, J_1= 1.0, J_2= 8.8 Hz, Ar-$\underline{H}$), 6.77 (t, 1H, J= 7.3 Hz, Ar-$\underline{H}$), 6.65 (dd, 2H, J_1= 1.0, J_2= 8.6 Hz, Ar-$\underline{H}$), 4.20-4.24 (m, 1H, -C$\underline{H}$-OH), 4.04 (dd, 1H, J_1= 4.1, J_2= 9.5 Hz, HO-CH-C$\underline{H}$-O-Ar), 4.00 (dd, 1H, J_1= 6.1, J_2= 9.5 Hz, HO-CH-C$\underline{H}$-O-Ar), 3.94 (bs, 1H, -O$\underline{H}$ or -N$\underline{H}$), 3.40 (dd, 1H, J_1= 4.3, J_2= 13.0 Hz, –NH-C$\underline{H}$-CH-OH), 3.26 (dd, 1H, J_1= 7.2, J_2= 13.0 Hz, –NH-C$\underline{H}$-CH-OH), 2.76 (bs, 1H, -O$\underline{H}$, or -N$\underline{H}$); ^{13}C-NMR: δ (ppm) 158.38, 148.03, 129.56, 129.29, 121.26, 118.03, 114.52, 113.26, 70.02, 68.76, 46.61.

1-(Benzylamino)-3-phenoxypropan-2-ol (Entry 15). m.p.: 71-72 °C; GC-MSD for $C_{16}H_{19}NO_2$ (mw: 257.34 g/mol): [M+1]$^+$ = 258, [M+29]$^+$ = 286, [M+41]$^+$ = 298, [M-17]$^+$ = 240; IR (cm^{-1}): 3266, 3003, 2938, 2853, 2716, 1600, 1586, 1497, 1464, 1454, 1363, 1339, 1304, 1294, 1247, 1211, 1158, 1115, 1068, 1043, 1022, 897, 882, 791, 749; ^{1}H-NMR: δ (ppm) 7.22-7.32 (m, 7H, Ar-$\underline{H}$), 6.93 (t, 1H, J= 7.3 Hz, Ar-$\underline{H}$), 6.88 (d, 2H, J= 7.9 Hz, Ar-$\underline{H}$), 4.04-4.08 (m, 1H, -C$\underline{H}$-OH), 3.95 (d, 1H, J= 9.9 Hz, HO-CH-C$\underline{H}$-O-Ar), 3.92 (d, 1H, J= 9.9 Hz, HO-CH-C$\underline{H}$-O-Ar), 3.82 (d, 1H, J= 13.2Hz, Ar-C$\underline{H}$-NH), 3.78 (d, 1H, J= 13.2 Hz, Ar-C$\underline{H}$-NH), 2.84 (dd, 1H, J_1= 3.9, J_2= 12.1 Hz, -NH-C$\underline{H}$-CH-OH), 2.75 (dd, 1H, J_1= 7.9, J_2= 12.1 Hz, -NH-C$\underline{H}$-CH-OH), 2.68 (bs, 2H, -N$\underline{H}$, -O$\underline{H}$); ^{13}C-NMR: δ (ppm) 158.56, 139.82, 129.43, 128.41, 128.07, 127.06, 120.96, 114.49, 70.38, 68.37, 53.78, 51.27.

1-(Phenylamino)-3-(o-tolyloxy)propan-2-ol (Entry 16). m.p.: 51-52 °C; GC-MSD for $C_{12}H_{19}NO_2$ (mw: 257.33 g/mol): [M+1]$^+$ = 258, [M+29]$^+$ = 286, [M+41]$^+$ = 298, [M-17]$^+$ = 240. IR (cm^{-1}): 3328, 2932, 1604, 1496, 1464, 1436, 1368, 1306, 1288, 1245, 1196, 1123, 1070, 1051, 1043, 842, 745; ^{1}H-NMR: δ (ppm) 7.12-7.20 (m, 4H, Ar-$\underline{H}$), 6.88 (t, 1H, J= 6.9 Hz, Ar-$\underline{H}$), 6.80 (d, 1H, J= 8.3 Hz, Ar-$\underline{H}$), 6.73 (t, 1H, J= 7.3 Hz, Ar-$\underline{H}$), 6.66 (d, 2H, J= 7.6 Hz, Ar-$\underline{H}$), 4.22-4.28 (m, 1H, -C$\underline{H}$-OH), 4.04 (dd, 2H, J_1= 4.5, J_2= 5.7 Hz, C$\underline{H}_2$-O-Ar), 4.00 (bs, 1H, , -O$\underline{H}$ or -N$\underline{H}$), 3.45 (dd, 1H, J_1= 4.3, J_2= 13.0 Hz, NH-C$\underline{H}$-

CH-OH), 3.29 (dd, 1H, J_1= 7.2, J_2= 13.0 Hz, NH-C$\underline{H}$-CH-OH), 2.59 (bs, 1H, -O$\underline{H}$ or -N$\underline{H}$), 2.25 (s, 3H, Ar-C$\underline{H}_3$); ^{13}C-NMR: δ (ppm) 156.41, 148.04, 130.83, 129.30, 126.92, 126.62, 121.00, 118.01, 113.25, 111.11, 70.06, 68.87, 46.77, 16.28.

1-(Benzylamino)-3-(o-tolyloxy)propan-2-ol (Entry 17). m.p.: 76-77 °C; GC-MSD for $C_{17}H_{21}NO_2$ (mw: 271.36 g/mol): [M+1]$^+$ = 272, [M+29]$^+$ = 300, [M+41]$^+$ = 312, [M-17]$^+$ = 255. IR (cm^{-1}): 3277, 3026, 2858, 2822, 1603, 1592, 1495, 1377, 1341, 1306, 1289, 1245, 1191, 1154, 1121, 1098, 1073, 1050, 1037, 1029, 995, 982, 935, 915, 888, 838, 767, 745, 737, 693; ^{1}H-NMR: δ (ppm) 7.29-7.33 (m, 4H, Ar-$\underline{H}$), 7.22-7.27 (m, 1H, Ar-$\underline{H}$), 7.10-7.14 (m, 2H, Ar-$\underline{H}$), 6.85 (t, 1H, J= 7.3 Hz, Ar-$\underline{H}$), 6.78 (d, 1H, J= 7.8 Hz, Ar-$\underline{H}$), 4.06-4.10 (m, 1H, -C$\underline{H}$-OH), 3.97 (dd, 1H, , J_1= 5.5, J_2= 9.5 Hz, C$\underline{H}$-O-Ar), 3.93 (dd, 1H, J_1= 5.0, J_2= 9.5 Hz, C$\underline{H}$-O-Ar), 3.84 (d, 1H, J= 13.2 Hz, Ar-C$\underline{H}$-NH), 3.79 (d, 1H, J= 13.2 Hz, Ar-C$\underline{H}$-NH, 2.89 (dd, 1H, J_1= 4.0, J_2= 12.2 Hz, NH-C$\underline{H}$-CH-OH), 2.80 (dd, 1H, J_1= 7.6, J_2= 12.2 Hz, NH-C$\underline{H}$-CH-OH), 2.56 (bs, 2H, -O$\underline{H}$, -N$\underline{H}$)), 2.19 (s, 3H, Ar-C$\underline{H}_3$); ^{13}C-NMR: δ (ppm) 156.62, 139.86, 130.65, 128.42, 128.08, 127.08, 126.77, 120.67, 111.08, 70.43, 68.47, 53.82, 51.38, 16.22.

1-(4-Methoxyphenoxy)-3-(phenylamino)propan-2-ol (Entry 18). m.p.: 69-71 °C; GC-MSD for $C_{16}H_{19}NO_3$ (mw: 273.33 g/mol): [M+1]$^+$ = 274, [M+29]$^+$ = 302, [M+41]$^+$ = 314, [M-17]$^+$ = 256; IR (cm^{-1}): 3377, 2998, 2926, 2830, 1607, 1526, 1503, 1459, 1438, 1292, 1264, 1228, 1218, 1180, 1153, 1125, 1113, 1036, 918, 878, 823, 749, 741; ^{1}H-NMR: δ (ppm) 7.17 (dd, 2H, J_1= 7.3, J_2= 8.6 Hz, Ar-$\underline{H}$), 6.80-6.85 (m, 4H, Ar-$\underline{H}$), 6.72 (t, 1H, J= 8.3 Hz, Ar-$\underline{H}$), 6.64 (d, 2H, J= 8.6 Hz, Ar-$\underline{H}$), 4.17-4.21 (m, 1H, -C$\underline{H}$-OH), 3.99 (dd, 2H, J_1= 4.1, J_2= 9.5 Hz, HO-CH-C$\underline{H}$-O-Ar and -O$\underline{H}$ or -N$\underline{H}$), 3.95 (dd, 1H, J_1= 8.1, J_2= 9.5 Hz, HO-CH-C$\underline{H}$-O-Ar), 3.73 (s, 3H, -OC$\underline{H}_3$), 3.38 (dd, 1H, J_1= 4.3, J_2= 12.9 Hz, –NH-C$\underline{H}$-CH-OH), 3.24 (dd, 1H, J_1= 7.2, J_2= 12.9 Hz, –NH-C$\underline{H}$-CH-OH), 2.80 (bs, 1H, -O$\underline{H}$ or -N$\underline{H}$); ^{13}C-NMR: δ (ppm) 154.16, 152.54, 148.05, 129.28, 117.96, 115.52, 114.69, 113.23, 70.83, 68.82, 55.69, 46.57.

1-(Benzylamino)-3-(4-methoxyphenoxy)propan-2-ol (Entry 19). m.p.: 94-95 °C; GC-MSD for $C_{12}H_{21}NO_3$ (mw: 287.36 g/mol): [M+1]$^+$ = 288, [M+29]$^+$ = 316, [M+41]$^+$ = 328, [M-17]$^+$ = 270; IR (cm^{-1}): 3288, 2905, 2828, 1509, 1506, 1466, 1455, 1444, 1345, 1291, 1232, 1107, 1077, 1068, 1049, 1013, 949, 906, 874, 836, 815, 753, 743, 699; ^{1}H-NMR: δ (ppm) 7.27-7.33 (m, 4H, Ar-$\underline{H}$), 7.22-7.26 (m, 1H, Ar-$\underline{H}$), 6.78-6.84 (m, 4H, Ar-$\underline{H}$), 4.02-4.06 (m, 1H, -C$\underline{H}$-OH), 3.90 (d, 2H, J= 5.2 Hz, C$\underline{H}_2$-O-Ar), 3.83 (d, 1H, J= 13.2 Hz, Ar-C$\underline{H}$-NH), 3.79 (d, 1H, J= 13.2 Hz, Ar-C$\underline{H}$-NH), 3.74 (s, 3H, -OC$\underline{H}_3$), 2.84 (dd, 1H, J_1= 3.9, J_2= 12.2 Hz, NH-C$\underline{H}$-CH-OH), 2.75 (dd, 1H, J_1= 7.8, J_2= 12.2 Hz, NH-C$\underline{H}$-CH-OH), 2.59 (bs, 2H, -O$\underline{H}$, -N$\underline{H}$); ^{13}C-NMR: δ (ppm) 153.97, 152.75, 139.84, 128.43, 128.06, 127.05, 115.48, 114.60, 71.20, 68.48, 55.66, 53.77, 51.24.

In view of the difficulties associated with the purification process, the minority products from entries 2, 4 and 10 were identified through gas chromatography coupled to a mass detector in the chemical ionization mode, which displayed different retention times, with the same molecular ions, and fragmentation patterns that were different respect to those of the majority products.

1-(Phenylamino)hexan-2-ol (Entry 2). [M+1]$^+$ = 194, [M+29]$^+$ = 222, [M+41]$^+$ = 234 m/z [M- OH]$^+$ = 176, [M- CH$_2$OH]$^+$ = 162.

1-(Pphenylamino)hex-5-en-2-ol (Entry 4). $[M+1]^+ = 192$, $[M+29]^+ = 220$, $[M+41]^+ = 232$. m/z $[M-OH]^+ = 174$, $[M-CH_2OH]^+ = 160$.

2-Phenyl-2-(phenylamino)ethanol (Entry 10). $[M+1]^+ = 214$, $[M+29]^+ = 242$, $[M+41]^+ = 254$. m/z $[M-OH]^+ = 196$. The $[M-31]^+ = [M-CH_2OH]^+ = 182$ fragment was not observed.

Acknowledgements

References and Notes

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