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ACTIVATION AND CHARACTERIZATION OF ALGERIAN KAOLINITE, NEW AND GREEN CATALYST FOR SYNTHESIS OF POLYSTYRENE AND POLY(1,3-DIOXOLANE)

Sarra Sabrina Aiche[1], Hodhaifa Derdar[1, 2, ✉], Zakaria Cherifi[1, 2],
Mohammed Belbachir[1], Rachid Meghabar[1]

Abstract. In the present work we have explored a new catalyst prepared with Algerian clay and a new method to synthesise polystyrene and poly(1,3-dioxolane). This technique consists of using Algerian modified clay (Kaolinite-H$^+$) as a green catalyst. Kaolinite-H$^+$ is a proton exchanged clay which is prepared through a simple exchange process. Synthesis experiments are performed in bulk. The polymerization of styrene in bulk leads to the yield of 83 % at room temperature with the reaction time of 3 h. Molecular weight of the obtained polystyrene is calculated by ^{1}H NMR and is about 2196 g/mol. Polymerization of (1,3-dioxolane) is carried out at room temperature with the reaction time of 3 h and polymerization yield of 91 %. The calculated molecular weight of the obtained poly(1,3-dioxolane) is about 573 g/mol. The structure of the obtained polymers is confirmed by FT-IR and ^{1}H NMR. The modified clay (Kaolinite-H$^+$) is characterized by FT-IR, XRD and SEM analysis.

Keywords: Kaolinite-H$^+$, Algerian clay, eco-catalyst, polystyrene, poly(1,3-dioxolane).

1. Introduction

The use of acid modified clay as a way to promote cationic polymerization has been the subject of numerous reports [1]. It provides a greener, less toxic and a more cost-effective alternative to the conventional cationic initiator such as metal based Lewis acids [2] and Bronsted acids [3]. Besides, these acid activated clays are more stable to the air and suffer less from oxidation trends [4].

[1] Laboratoire de Chimie des Polymères, Département de Chimie, FSEA, Université Oran1
Ahmed Benbella BP N° 1524 El M'Naouar, 31000 Oran, Algeria
[2] Centre de Recherche Scientifique et Technique en Analyses Physico-Chimiques (CRAPC),
BP 10 384, Siège ex-Pasna Zone Industrielle, Bou-Ismail CP 42004, Tipaza, Algeria
✉ hodhaifa-27@outlook.fr

The Smectites clays were known for a long time for their significant ability to catalyze efficiently various organic reactions [5]. Gómez-Sanz *et al.* [6] reported a work emphasizing the role of raw saponites and montmorillionites as acidic catalyst. They demonstrated that acid clay was an efficient and recyclable catalyst for a Mannish reaction to synthesis *β*-amino ketones.

Although the preparation of clay/polymer nanocomposites was a well-known process that has been wildly described [7], the use of clays as initiators for polymerization processes on the other hand was limited. Nevertheless, the work of Wang *et al.* [8] on the polymerization of phenolic compounds using montmorillonite, illite and kaolinite is worth mentioning for its pioneer aspect. It was only in the early 20th, thanks to the work of Belbachir *et al.* [9], that the use of clays as a way to promote cationic polymerizations became a common trend. During the last two decades, there have been several reports describing the use of acid activated montmorillonite as a cationic initiator for different polymerization and copolymerization processes of numerous linear and cyclic monomers including isobutylene [10], styrene [11], *N*-vinyl-2-pyrrolidone [12], *β*-pinene [13], glycidol [14], and dodecamethylcyclohexasiloxane [15].

Kaolinite, which is the main mineral contained in kaolin deposit clays, is also known to have various applications such as nanofillers for the preparation of polymer nanocomposites [16], fillers in the rubber and paper industries [17], pigments in paint [18], and ceramics [19]. However, we did notice through our researches that the use of kaolinite as catalyst was somehow limited. Its largest use was as catalyst subtracts in catalytic cracking processes [20]. In fact, kaolinite is considered as less reactive in comparison with other clays families such as smectites, palygorskite and sepiolite [21]. There are only few reported works on the use of kaolinite as a catalyst to promote polymerization [22]. Wang *et al.* [23] investigated the effect of kaolinite in the formation of hydroquinone-derived polymers. Zhao *et al.* [24] also studied the catalytic aspect of kaolinite in the preparation of polystyrene.

Generally, almost all the catalysts used for the preparation of polymers present great environmental problems, as they are corrosive. The main drawbacks of the application of zinc and silver compounds as well as Lewis acids are the difficulty to handle and complicated separation. Moreover, zinc or silver compounds require environmental reconditioning [25]. In this work, we have described the preparation of new acid activated kaolinite clay as a potential cationic initiator for the polymerization of both linear and cyclic monomers. The catalytic activity of this clay was investigated through the synthesis of two different polymers: polystyrene and poly(1,3-dioxolane). The polymerizations were carried out under mild conditions with respect to the green chemistry principles.

By examining the literature, we found that the studies of the catalytic properties of Algerian kaolinite for polymerization of styrene and 1,3-dioxolane are practically nonexistent. The main purpose of this work is to study the properties of the modified kaolinite as a new and non-toxic catalyst for cationic polymerization of both linear and cyclic monomers. Kaolinite is preferred for its many advantages such as very low purchase price compared to other catalysts and easy removal of the reaction mixture, regenerated by heating to the temperature above 393 K. FT-IR, XRD and SEM analysis confirm the acid activation of kaolinite. ^{1}H NMR and FT-IR analysis show that the polymerization of styrene and 1,3-dioxolane is successfully with Kaolinite-H$^+$.

2. Experimental

2.1. Materials

Styrene (98 %), 1,3-dioxolane (97 %), methanol (CH$_3$OH, 99.9 %) and dichloromethane (CH$_2$Cl$_2$, 99.8 %) were supplied by Sigma Aldrich and used as received. Kaolinite (Algerian clay) was supplied in the raw state, Tounane (Algeria).

2.2. Purification of Kaolinite

The purification proceeds by washing the raw kaolinite with distilled water. The kaolinite is ground first and then sieved ($\delta < 0.08$ mm). 30 g of kaolinite are dispersed in 1 liter of distilled water (2 wt %) and kept under magnetic stirring for 24 h at room temperature.

After stopping the stirring the suspension is decanted for a few minutes and filtered. This preliminary treatment allows the removal of water-soluble materials such as salts. The clay thus recovered is dried at 393 K, then ground in a mortar, sieved ($\delta < 0.08$ mm) and stored in a desiccator under vacuum.

2.3. Preparation of Kaolinite-H$^+$

Kaolinite-H$^+$ is activated with a chloridric acid solution (HCl) to give a kaolinite exchanged with protons, called Kaol-H$^+$. In an Erlenmeyer flask, crushed raw kaolinite (30 g) is dispersed in a volume of distilled water (500 ml). The mixture is stirred for 20 min at room temperature. Then, a solution of chloridric acid (0.5M) is added. The solution thus obtained is maintained for 120 min under stirring. The mineral is filtered off and washed several times with distilled water up to pH 7. After filtration, Kaol-H$^+$ is dried in an oven for 2 h, at 378 K and is then crushed; the same experimental protocol is performed with 6M and 12M HCl. The composition of kaolinite is given in Table 1 and the results show that the amount of Fe$_2$O$_3$ is very important in the KT which gives it the red color except in the Kaolinite of Portugal. The amount of SiO$_2$ is almost the same in all kaolinites. The amount of Al$_2$O$_3$ is almost the half. We also noted that there are traces of MgO and K$_2$O in all kaolinites except in the Kaolinite of Portugal. Its structure is established by FT-IR, XRD and SEM analysis.
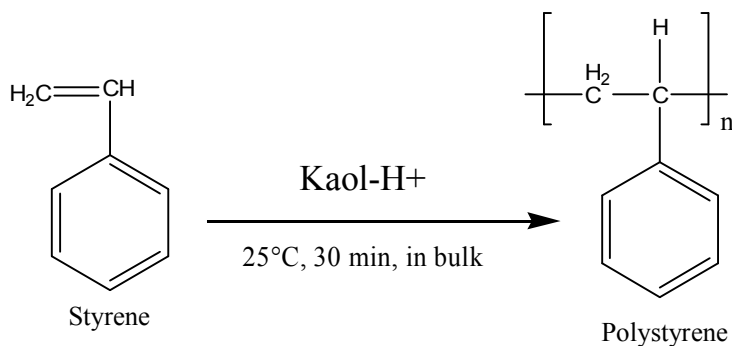
2.4. Polymerization Procedure

The reactions of polymerization of styrene and 1,3-dioxolane (Schemes 1 and 2) are carried out, in bulk using Kaol-H$^+$ as a catalyst. In a flask containing 2 g of styrene, we added 5 wt % of Kaol-H$^+$, which is previously dried for 30 min in the oven at 378 K. The mixture is kept under stirring for 2 h at room temperature (298 K), the reaction mixture is dissolved in CH$_2$Cl$_2$, filtered, precipitated in cold methanol and dried in vacuum over night. The obtained polymer is a solid product. Then, the same experimental protocol is performed with 5 wt % of Kaol-H$^+$, but this time to polymerize 1,3-dioxolane.

Table 1

Comparison of elementary compositions of kaolinite

Elements	Fe$_2$O$_3$	SiO$_2$	Al$_2$O$_3$	MgO	K$_2$O	LOI
Algerian Kaolinite	12.96	45.87	16.59	0.59	1.83	22.16
Kaolin of Tamazert [26]	0.98	57.7	37.98	0.33	1.07	1.94
Tunisian Kaolinite [27]	1.63	58.49	22.32	0.62	1.42	10.94
Kaolin of India [28]	0.86	45.81	37.72	0.32	0.03	13.56
Kaolin of Brazil [29]	0.84	45.56	36.63	0.04	0.10	16.48
Kaolin of Portugal [30]	0	53.11	46.37	0	0	0.52

Scheme 1. Polymerization of styrene catalyzed by Kaol-H[+]
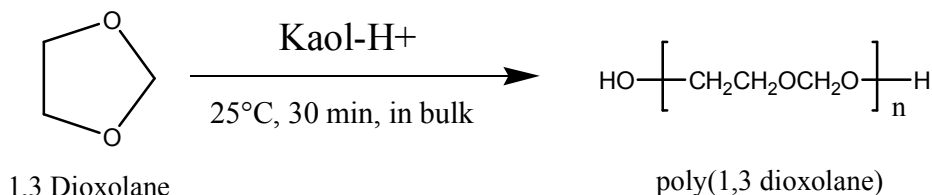
Scheme 2. Polymerization of 1,3-dioxolane catalyzed by Kaol-H[+]

Regarding the kinetic study, the same procedure described above was repeated by changing the percentage of the catalyst and the time of the reaction, in order to find the optimal reaction conditions.

2.5. Characterization

The X-Ray powder diffraction profiles for pressed powder samples were recorded on a D8 Discover Bruker diffractometer using Cu-Kα radiation ($\lambda = 1.5418$ Å). The surface morphology of the modified clay was observed by FEG-SEM on a JEOL 7001F electron microscopy. FTIR absorption spectra were recorded on an Alpha Bruker FTIR spectrometer. NMR is a very powerful analytical method for the elucidation of chemical structures. ^{1}H NMR spectra are recorded on a Bruker-Avance 300MHZ apparatus in Deuterated Chloroform.

3. Results and Discussion

3.1. Characterization of Kaolinite-H[+]

Fig. 1 shows the FT-IR spectrum of raw kaolinite and kaolinite washed with distilled water and dried at 393 K. We noted a change in two spectrums at the bands of 3397 and 3697 cm[-1], which correspond to the OH bands vibration. These bands disappear during the drying of kaolinite at 393 K indicating the disappearance of the OH molecules.

The bands at 3397 and 3697 cm[-1] correspond to the frequency of O-H stretching vibrations and the peak at 3697 cm[-1] is attributed to the elongation of the inner surface hydroxyl groups. The band located at 3622 cm[-1] corresponds to the elongation of the internal hydroxyl group of kaolinite. The lower frequency bands, appearing around 1400 cm[-1], correspond to the Si-O-Si elongation vibrations, the band that appears at 911 cm[-1] corresponds to the vibrations of the Al_2-OH, and the band located at 795 cm[-1] can be attributed to the asymmetric and symmetric elongation vibration of the Si-O-Si group. All these expected absorption bands confirm that the structure of the compound is in a good agreement with vibration values obtained from the literature [31].

The X-ray diffractograms of raw Kaolinite and Kaolinite-H[+] are shown in Fig. 2. The Figure shows a slight change in the interlayer distance according to Table 2 after the quartz extraction. We also note that in the diffractogram (A) the intensity of the quartz is 3232 counts while in the diffractogramme (B) the intensity went down to 637counts.

The SEM images of raw-Kaol and modified Kaolinite (Kaol-H[+]) are shown in Fig. 3. These images are very similar to those obtained by M. Hernandez [32]. The SEM images showed that the morphology of kaolinite was retained after modification by the HCl acid. The particle sizes of all samples are about 1–10 μm. These particles exhibited bulky flakes and aggregated disorderly form confirming the layered structure of clay, indeed the modified clays are expected to have a similar morphology.
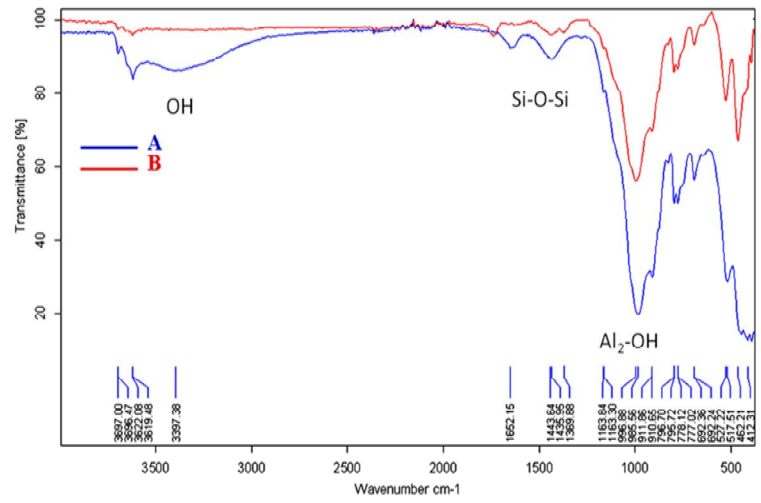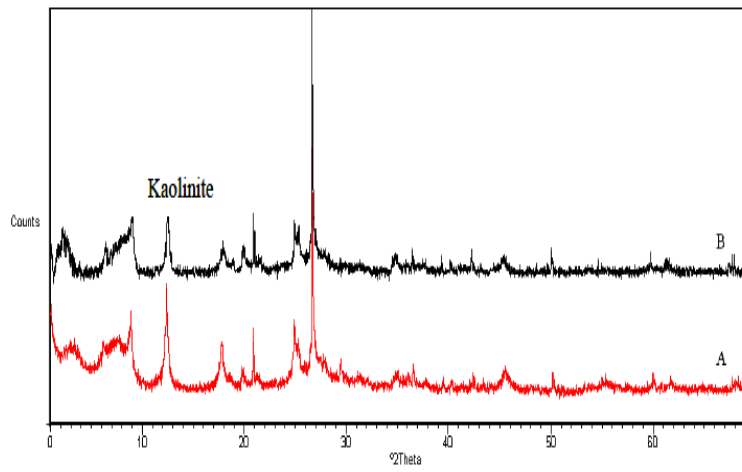
Fig. 1. FT-IR spectra of raw-Kaol (A) and Kaol-H$^+$ (B)

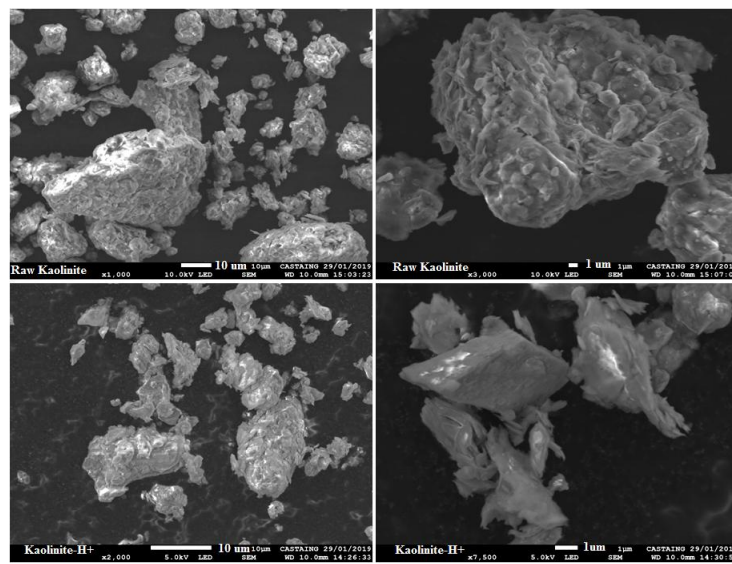**Fig. 2.** X-ray diffractograms of raw-Kaol (B) and Kaol-H$^+$ 0.25M (A)

Fig. 3. SEM images of raw-Kaol and Kaol-H$^+$

Table 2

Change in interlayer distances before and after acid activation

	A	B
Halloysite 10 Å	9.98	10.07
Halloysite 7 Å	4.25	4.48
Kaolinite	7.13	7.20
Quartz	3.38	3.35

3.2. Characterization of the Obtained Polymers (Polystyrene and Poly(1,3-Dioxolane))

3.2.1. Fourier Transform Infrared Spectroscopy (FT-IR)

FT-IR spectra of polystyrene and poly(1,3-dioxolane) are shown in Figs. 4 and 5, respectively. The FT-IR spectrum of polystyrene (Fig. 4) shows the following characteristic bands: the bands at 2848 and 2919 cm^{-1} correspond to the aliphatic C–H vibration frequencies and the band at 3024 cm^{-1} corresponds to the aromatics C–H elongation vibrations. The peaks at 690 and 760 cm^{-1} correspond to the deformation of a substituted aromatic ring and the disappearance of the 1630 cm^{-1} band corresponding to the frequency of elongation vibrations of the vinyl double bond of styrene, shows that the polymerization of styrene with Kaol-H$^+$ has succeeded; the same result is obtained by N.Bensaada *et al.* [33].

FT-IR spectrum of poly(1,3-dioxolane) shown in Fig. 5 is characterized by different vibration bands; the bands between 2881 and 2956 cm^{-1} correspond to the vibrations of C–H of methyl group. The band at 1450 cm^{-1} is assigned to the vibrations of C–C. The bands at 1018 and 1300 cm^{-1} are attributed to C–O groups of ether functions. Similar results were obtained by M. Belbachir *et al.* [34].
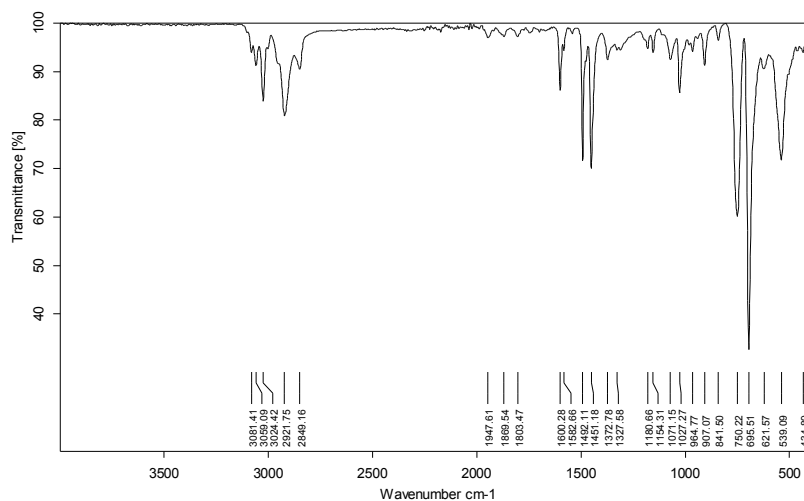

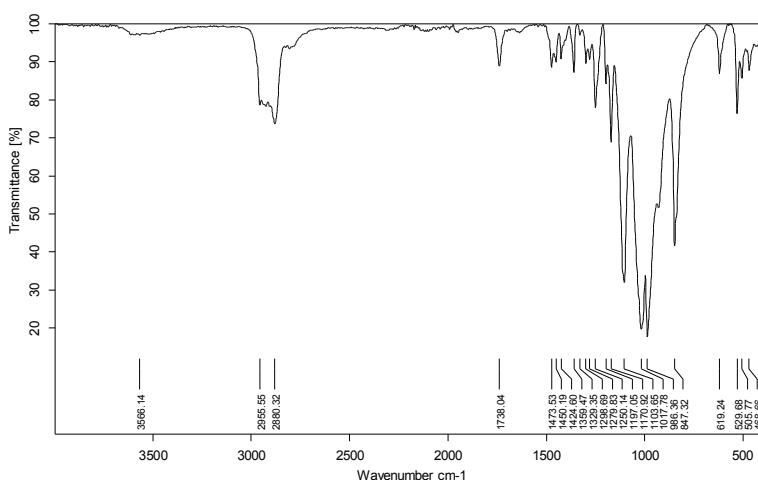**Fig. 4.** FT-IR spectrum of the obtained polystyrene

Fig. 5. FT-IR spectrum of the obtained poly(1,3-dioxolane)

3.2.2. Nuclear Magnetic Resonance of Proton (^{1}H-NMR)

NMR spectrum of the obtained polystyrene is shown in Fig. 6. ^{1}H NMR spectrum of polymer was recorded in CDCl$_3$ using a Bruker 300 MHz apparatus at 298 K and gives the following information:

The ^{1}H NMR spectrum confirms the structure of the polymer obtained in this study. The signals between 6.6 and 7.3 ppm are attributed to the aromatic protons. The peaks at 5 and 6 ppm are the characteristic resonance of the protons resulted by the terminal double bond (–C=CH) of the main chain as large signal in the range of 1.5–2 ppm and the terminal methyl groups at 1.2 ppm. The ^{1}H NMR spectrum of the obtained polystyrene is similar to that of polystyrene obtained with Maghnite-H$^+$

[35], which clearly shows that the synthesis of polystyrene is successful with Kaol-H$^+$.

^{1}H NMR spectra of poly(1,3-dioxolane) is shown in Fig. 7. The polymerization is carried out in bulk at 298 K for 30 min, the peak "b" at 4.7 ppm and the peak "a" at 3.71 ppm correspond to protons in methyl group (C–H). The peak at 2 ppm is a characteristic resonance of the proton resulted by the terminal hydroxyl group (OH).

The molecular weight of the obtained polymers determined by ^{1}H NMR spectrum is reported in Table 3.

Table 3

Molecular weight of the obtained polymers

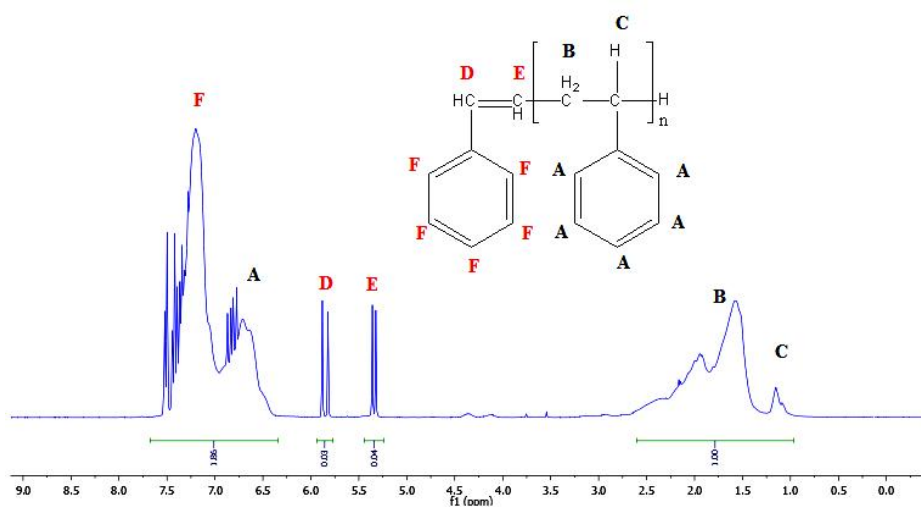| Product	Molecular weight, g·mol^{-1}
Polystyrene	2196
Poly(1,3-dioxolane)	573

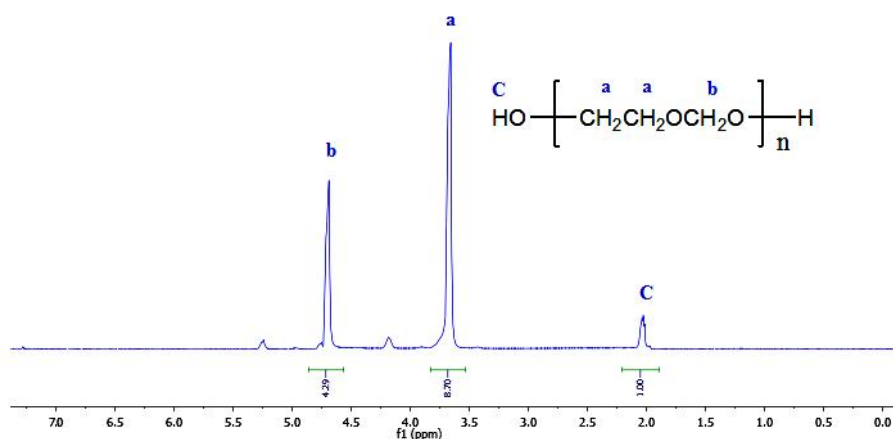**Fig. 6.** ^{1}H NMR spectrum of the obtained polystyrene

Fig. 7. ^{1}H NMR spectrum of the obtained poly(1,3-dioxolane)

3.3. Determination of Optimal Reaction Conditions

The objective of this part is to study the kinetic of the polymerization of styrene and 1,3-dioxolane with Kaol-H$^+$, which consists in varying separately different parameters including the quantity of the catalyst and the reaction time, in order to know their influence on the yield of the obtained polymers and to find the optimum conditions of the reaction. The % yield of the obtained polymers is calculated by a simple weighing according to the following equation: yield (%) = $(M_0/M_1) \cdot 100$, where M_0 is a weight of the obtained polymer; M_1 is a weight of the initial monomer.

3.3.1. Effect of Kaol-H$^+$ amount on the yields of polystyrene and poly(1,3-dioxolane)

Fig. 8 shows the effect of the catalyst amount on the yield of the obtained polymers. The polymerization reactions were carried out at 298 K for 30 min using various amounts of Kaol-H$^+$ (2, 5, 7, 10, and 15 wt %), the reactions were carried out in bulk. Fig. 8 shows that the yield of polymers increased with the amount of Kaol-H$^+$. The yield increases according to the quantity of Kaol-H$^+$ up to the optimal point, which corresponds to 15 wt %. This behavior is explained by the increase of active sites available in Kaol-H$^+$ responsible for the initiation and acceleration of the reaction until the saturation of these sites. After the reaction the solid catalyst is totally regenerated by simple filtration; similar results were obtained by Belbachir and coworkers using Maghnite-H$^+$ as a catalyst [36].
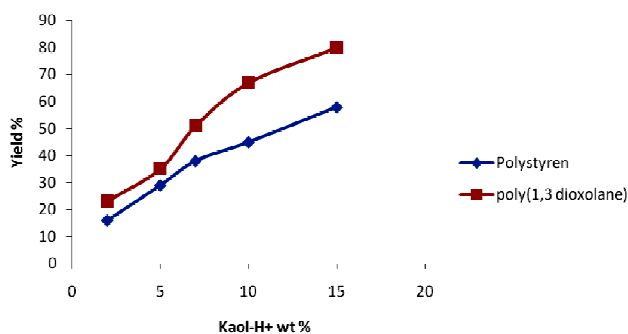

Fig. 8. Effect of the Kaol-H$^+$ amount on the yield of the obtained polymers

3.3.2. Effect of time on the yield of the obtained polymers

Fig. 9 shows the yield of the polymers *versus* synthesis time for polystyrene and poly(1,3-dioxolane) using Kaol-H$^+$ as the catalyst. As the Figure shows, at the end of 3 h, reaction takes place quickly, reaching the best yields (83 % for polystyrene and 91 % for poly(1,3 dioxolane)) in the presence of 10 wt %wt of Kaol-H$^+$ at 298 K. After this time, the reaction slows down gradually, and the yield becomes almost constant. This behavior is explained by the nature of the reaction conducted in bulk.
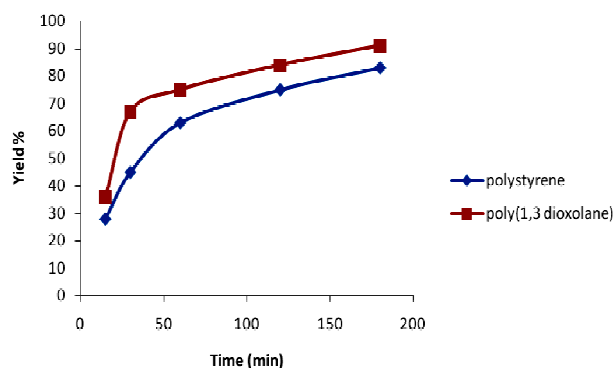

Fig. 9. Effect of the reaction time on the yield of the obtained polymers

4. Conclusions

In the present work, we have developed a new and green catalyst to polymerize vinylic and cyclic monomers. These materials were prepared using an Algerian clay (Kaolinite-H$^+$), that could be used as green and eco-catalyst. The synthesis of polystyrene and poly(1,3-dioxolane) over the Algerian Kaolinite provides excellent results in bulk, at room temperature and in one step. The influencing factors on the synthesis reaction were studied, and the optimal reaction conditions were obtained and the yield achieved its maximum 83 % for polystyrene and 91 % for poly(1,3-dioxolane) with 15 wt % of Kaolinite-H$^+$ at room temperature for 3 h, according to increase in acidic active centers. The resulting products were perfectly characterized by FT-IR and ^{1}H NMR. Kaolinite-H$^+$ is an efficient catalyst, cheap and environmentally friendly. Its use in the polymerization of vinylic and cyclic monomers leads to good results.

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АКТИВАЦІЯ ТА ХАРАКТЕРИСТИКА АЛЖИРСЬКОГО КАОЛІНІТУ, НОВОГО І ЕКОБЕЗПЕЧНОГО КАТАЛІЗАТОРА СИНТЕЗУ ПОЛІСТИРЕНУ ТА ПОЛІ(1, 3-ДІОКСОЛАНУ)

Анотація. *Досліджено новий каталізатор, приготований з алжирської глини, та розроблено новий метод синтезу полістиролу та полі(1,3-діоксолану). Показано, що модифіковану алжирську глину (Каолініт-H^+) можна використовувати як екокаталізатор. Каолініт-H^+ являє собою протонно-обмінену глину, приготовлену звичайною реакцією обміну. Встановлено, що полімеризація стирену в масі за кімнатної температури та часі реакції 3 години дає можливість одержувати продукт з виходом 83 %. З використанням ^1H ЯМР визначена молекулярна маса отриманого полістирену, яка становить приблизно 2196 г/моль. При полімеризації (1,3-діоксолану) за кімнатної температури та часі реакції 3 год отримано продукт з виходом 91%. Розрахована молекулярна маса отриманого полі(1,3-діоксолана) становить приблизно 573 г/моль. Структуру отриманих полімерів підтверджено Фур'є-спектроскопією, ^1H ЯМР, рентгеноструктурним аналізом та скануючої електронною мікроскопією.*

Ключові слова: *каолініт-H^+, алжирська глина, екокаталізатор, полістирен, полі(1,3-діоксолан).*