

# Theoretical and Experimental Study of the Chemical Modification of Poly(epichlorohydrin) by Grafting Menthol<sup>†</sup>

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**Abstract:** Functional polymers, a significant class of compounds with diverse applications, are synthesized by incorporating functional groups into polymer matrices. Poly(epichlorohydrin) (PECH) is a pivotal polymer due to its reactive chloromethyl group, which readily enables its transformation into various functional polymers. Our study is focused on theoretically unraveling the mechanism behind modifying PECH through menthol grafting. We optimized and characterized various stationary states using density functional theory (DFT) at the B3LYP/6-311++G\*\* level. These calculations were conducted in both the gas phase and a solution environment (THF). To anticipate the specific sites within our polymer that would be subject to grafting, we calculated various reactivity indices at the same computational level.

**Keywords:** poly(epichlorohydrin); chloromethyl; grafting; density functional theory

## 1. Introduction

Functional polymers are produced by the integration of the identical or different functional groups into a polymer matrix. These polymers have attracted a lot of interest due to their numerous applications in recent years, such as in electrical [1,2] and electronic science [3,4], biotechnology [5,6], biochemistry [7,8], biomedical [9–11] (carriers of active ingredients), as well as in the agricultural field [12] (biocidal functions).

They constitute an important class of compounds with great activity in different fields. Indeed, the control of their structures from the point of view of their functionality has recently aroused great enthusiasm. Various applications, such as textiles, automobiles, optics, and drug molecules, illustrate the great versatility that these materials offer in booming fields. The ability to polymerize heterocyclic monomers essentially depends on the size of the ring, the nature of the heteroatom which contains it, as well as the nature of the substituent around the rings. Indeed, several studies have been carried out in this context [13–15]. It is important to modify the structure of a polymer according to tailor-made characteristics designed for target applications. Most research efforts have been reserved for attempts to transform functional polymers into products for the environmental and biological field. This technique has experienced very significant development in recent years, particularly in the field of industry; we can cite, for example, cellulose [16], which, after chemical modification, can lead to a large class of polymers. This chemical modification of polymers presents essential advantages in the world of polymers, such as the immobilization of macromolecular chains on polymer surfaces, the adjustment of the polarity of polymers, and the preparation of three-dimensional networks.

These polymers obtained after chemical modification, either by substitution reactions [17], elimination [18], reduction [19], oxidation [20], alkylation [21], or grafting [22],



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can be classified as monofunctional polymers (comprising a functional group at the end of the chain) or difunctional (telechelic) polymers (comprising functional groups at both ends of the polymer), or even multifunctional polymers (comprising functional groups, identical or different, all along its polymer chain).

In this context, our contribution is in fact twofold: on the one hand, experimental: the preparation of a three-membered heterocyclic polymer poly(epichlorohydrin), as a precursor polymer, by the cationic method by an activated monomer, followed by a chemical modification of this, and lastly by the technique of grafting with menthol; on the other hand, a theoretical study: the elucidation of the reaction mechanism of this chemical modification by reproducing, in the same condition, as best as possible, the obtained experimental results.

## 2. Methodology of Calculations

All theoretical calculations were carried out using the Gaussian 09 program [23] with the (DFT) method [24,25] at the B3LYP/6-311++G\*\* level [26]. An IRC calculation [27,28] was carried out. Given that the reaction occurs experimentally in an aprotic polar solvent (THF), the effect of the latter was taken into account implicitly in our calculations using Tomasi's PCM [29]. In order to understand the chemical reactivity trends of the reactants, reactivity indices, such as the chemical potential  $\mu$  and chemical hardness  $\eta$  for each reacting species, have been calculated at the same level of theory [30].

## 3. Results and Discussion

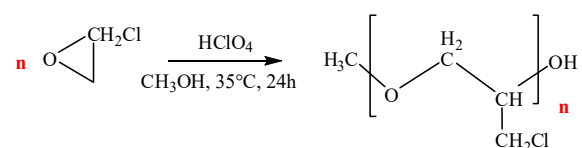
### 3.1. Experimental Study

**Materials:** Epichlorohydrin (ECH) monomer was purchased from "Sigma-Aldrich" and distilled under reduced pressure; methanol was distilled on the day of experiment; perchloric acid, menthol.

**Measurements:** FT-IR spectra were obtained on ALPHA FT-IR mark "Bruker", bearing the module "Platinumdiamond ATR single reflection ATR", at room temperature in the range of 400–4000  $\text{cm}^{-1}$ .

### 3.2. Synthesis of Poly(epichlorohydrin) (PECH)

The cationic mass polymerization by the ring-opening of epichlorohydrin was carried out by an activated monomer; the reaction was initiated by perchloric acid ( $\text{HClO}_4$ ) in the presence of methanol ( $\text{CH}_3\text{OH}$ ) [31] at a temperature of 35 °C under a nitrogen atmosphere and magnetic stirring for 24 h, according to the reaction Scheme 1.



**Scheme 1.** Reaction scheme of the cationic polymerization of epichlorohydrin by the activated monomer.

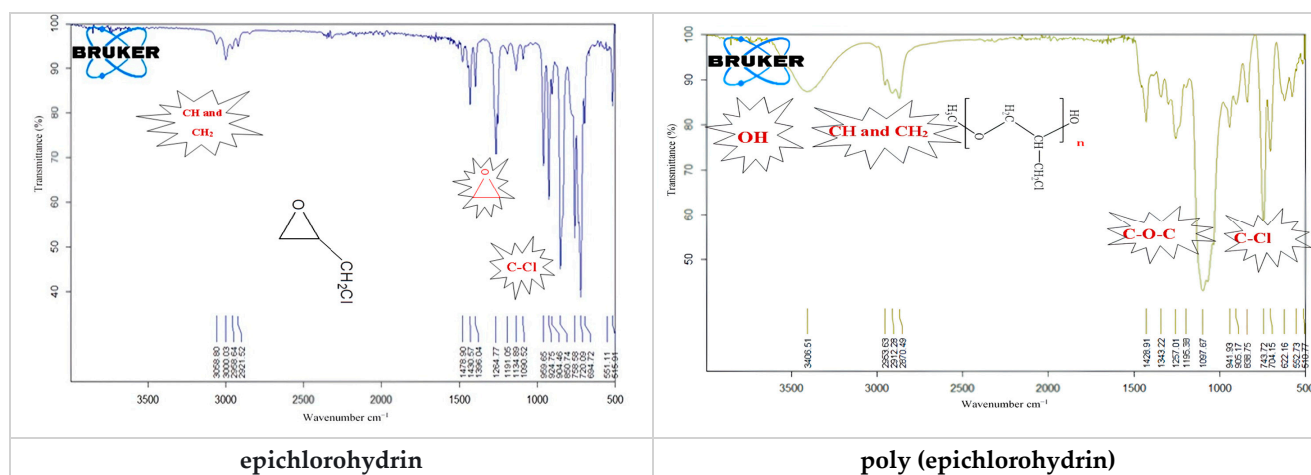
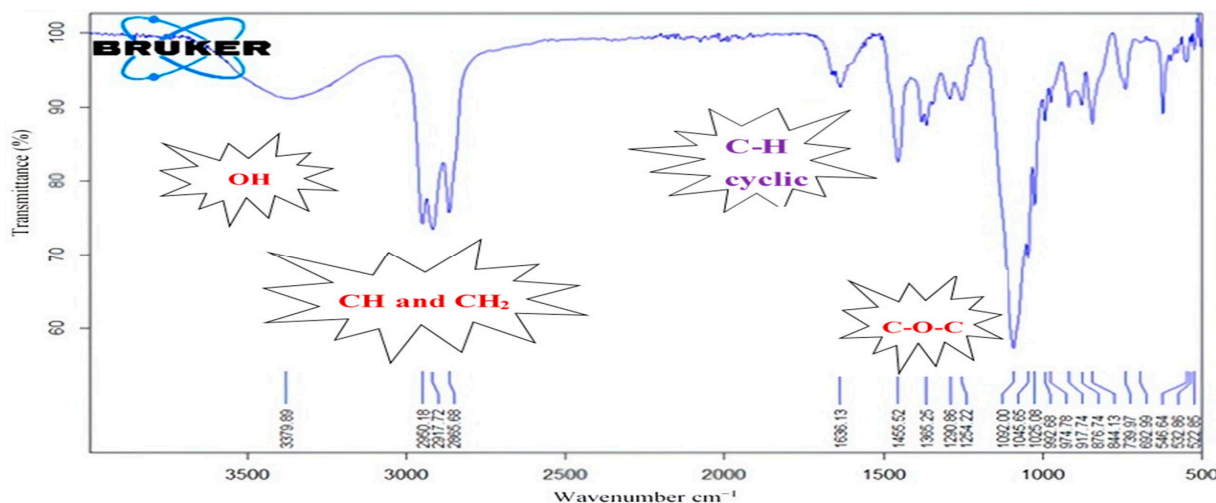
The cationic polymerization reaction was carried out in a three-necked distilling flask connected to a nitrogen supply. In order to prevent the solvent from being carried away by the nitrogen flow, a refrigerant was used to cool and condense the solvent vapors and to avoid any variation in the concentration of the reaction medium. The reaction was maintained under magnetic stirring and at a fixed temperature. Into the three-necked distilling flask, we introduced, successively, the methanol, the epichlorohydrin, and the perchloric acid; the latter was added drop-by-drop. At the end of the reaction, the crude product was precipitated in cyclohexane at room temperature. The polymer was obtained with a gross yield by weight of 92%. The experimental conditions of the ring-opening polymerization are summarized in Table 1.

**Table 1.** Experimental conditions and yield by weight of the synthesis of poly(epichlorohydrin) at T = 35 °C in 24 h.

Reagents	n (mol)	V (mL)	C (mol/L)	Weight (%)
ECH	0.127	10	0.0127	92
HClO <sub>4</sub>	0.0002	0.0120	0.1600	
MeOH	0.004	0.23	0.017	

### 3.3. Characterization of the Obtained Polymer

The FTIR spectrum of the monomer and the polymer obtained are, respectively, represented in Figures 1 and 2.

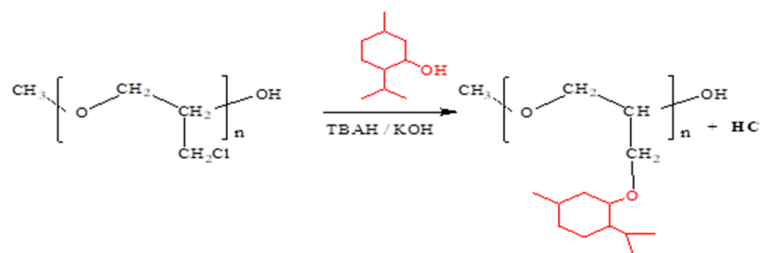
**Figure 1.** FTIR spectrum.**Figure 2.** IR spectrum of poly(epichlorohydrin) modified by menthol grafting.

The FTIR spectrum of the monomer epichlorohydrin illustrated in Figure 1 shows the main characteristic bands; in this case, an intense band at  $904\text{ cm}^{-1}$ , characteristic of oxirane, and an intense band at  $720\text{ cm}^{-1}$ , which characterizes the C-Cl bond. Furthermore, the FTIR absorption spectrum of the synthesized polymer reveals the presence of the main bands, which characterize the expected polymer. The FTIR spectrum (poly (epichlorohydrin)), allows us to first observe the disappearance of the characteristic band of oxiranes, located at  $904\text{ cm}^{-1}$ , and the appearance of a new absorption band at  $1097\text{ cm}^{-1}$ , characteristic of the ether function (C-O-C), and an elongation band located at  $3406\text{ cm}^{-1}$ , characteristic

of the hydroxyl groups (OH) located at the end of the homopolymer chain, and those of the adsorbed water. These results allow us to assume that the cationic ring-opening polymerization in the presence of methanol leads to the formation of poly(epichlorohydrin) with a hydroxyl end.

### 3.4. Chemical Modification of Poly(epichlorohydrin) by Grafting Menthol

The preparation of multifunctional polymers can be carried out by the chemical modification of the homopolymers [32,33]. These multifunctional polymers can serve as carriers for the active ingredients [34]. For our part, we tried to graft the active ingredients, such as menthol, a biodegradable natural product, onto poly(epichlorohydrin) (Scheme 2).



**Scheme 2.** Reaction scheme of the chemical modification of poly(epichlorohydrin) by menthol grafting.

The chemical modification of poly(epichlorohydrin) was carried out using the phase-transfer method. The homopolymer was dissolved in THF; at 25 °C, it was brought into contact with the menthol and an aqueous solution of KOH and TBAH, with magnetic stirring. The experimental conditions for this reaction are summarized in Table 2.

**Table 2.** Experimental conditions for the chemical modification of poly(epichlorohydrin).

Reagents	m (mg)	V (mL)	Weight (%)
PECH	1.28	--	62
MENTHOL	0.2	--	
KOH	28.05	20	
TBAH	0.43	--	
THF	--	20	

### 3.5. Characterization of the Obtained Polymer

The product obtained was characterized by IR absorption spectroscopy. The FTIR spectrum is illustrated in Figure 2.

From Figure 2, we notice the disappearance of the characteristic band of the  $\text{CH}_2\text{Cl}$  group observed in the FTIR spectrum of the homopolymer at  $743\text{ cm}^{-1}$ . On the other hand, we note the appearance of a new absorption band at  $1455\text{ cm}^{-1}$ , which characterizes the deformation of the cyclic C-H bonds belonging to the menthol cycle grafted onto the poly(epichlorohydrin), as well as the vibration of the C-O-C bond at  $1092\text{ cm}^{-1}$ .

This result allows us to conclude that the menthol was grafted onto the poly(epichlorohydrin) homopolymer using the phase-transfer method, thus leading to the formation of the multifunctional polymer.

### 3.6. Prediction of the Nucleophilic and Electrophilic Character of the Reagents

The reaction is governed by the LUMO control, since the LUMO gap is smaller ( $0.2792\text{ u.a.}$ ) than the HOMO gap ( $0.3478\text{ u.a.}$ ). Therefore, the interaction to be considered as a priority is that which develops between the HOMO of menthol (nucleophile) and the LUMO of the homopolymer (electrophile).

The electronic chemical potentials  $\mu$ , hardness  $\eta$ , electrophilicity indices  $\omega$ , and nucleophilicity indices  $N$  of the reagents are presented in Table 3.

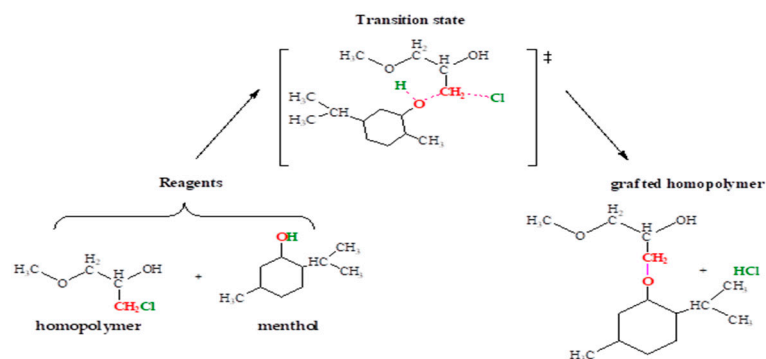
**Table 3.** Energy of the HOMO and LUMO frontier orbitals ( $E_{\text{HOMO}}$  and  $E_{\text{LUMO}}$ ), chemical potential ( $\mu$ ), hardness ( $\eta$ ), and electrophilicity ( $\omega$ ) (in eV) calculated at B3LYP/6-311++G\*\*.

Reagents	$E_{\text{HOMO}}$	$E_{\text{LUMO}}$	$\mu$	$\eta$	$\omega$	$N$
Homopolymer	−0.2726	0.0234	−7.0992	8.0547	3.1285	−17.6535
Menthol	−0.25588	0.0752	−5.9387	9.0100	1.9571	−17.1783

The results presented in Table 3 show that the chemical potential  $\mu$  of the homopolymer (−7.0992 eV) is lower than that of the menthol, implying that charge transfer will take place from the menthol to the homopolymer, and the menthol is less electrophilic than the monomer. In other words, the menthol is more nucleophilic than the homopolymer.

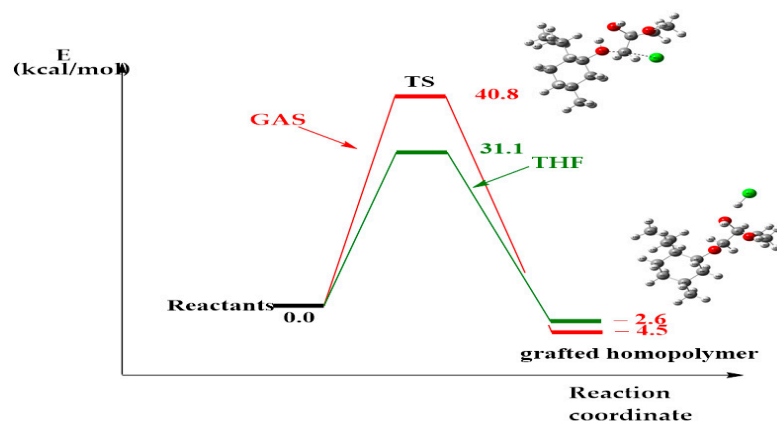
### 3.7. Energy Profile of the Reaction

A nucleophilic substitution reaction has been experimentally proposed for this reaction of the chemical modification of the polymer poly(epichlorohydrin) (PECH) by menthol. However, we attempted to determine by theoretical means the order of this reaction. We were first concerned with determining the transition state between the homopolymer and menthol (Scheme 3).



**Scheme 3.** Reaction path of the reaction of the chemical modification of (PECH) by the grafting of menthol. Double dagger ( $\ddagger$ ) represents that the reaction taking place is a transition state.

The energy calculation of the transition state ( $E$ ) and the activation barrier ( $E_a$ ) was carried out at the B3LYP/6-311++G\*\* level. The results are summarized in Figure 3.



**Figure 3.** Energy diagram of the chemical modification of the homopolymer modified by menthol in THF at the B3LYP/6-311++G\*\* level.

The reaction rate is notably influenced by the solvent, as evidenced by a substantial reduction in the activation energy barrier of 10 kcal/mol when comparing tetrahydrofuran (THF) to the gas phase. This reduction signifies a significant acceleration of the reaction in the presence of the solvent.

The comparison of calculated free enthalpies further indicates that the chemical modification of the homopolymer grafted by menthol is more favorable in the chosen solvent, THF. This thermodynamic favorability implies a greater likelihood of the reaction proceeding to completion. The preference for the SN2 mechanism, as suggested by our calculations, aligns with the solvent's role in facilitating the reaction.

#### 4. Conclusions

Cationic polymerization by ring-opening allowed us to synthesize the homopolymer of epichlorohydrin. For this, we adopted the activated monomer process to minimize transfer reactions. The polymerization reaction was carried out in bulk. Furthermore, the homopolymer of poly(epichlorohydrin) served as a precursor for the synthesis of modified poly(epichlorohydrin). The chemical modification of the homopolymer was carried out by eventually substituting chlorine, which exists along the polymer chain, with menthol. The homopolymer as well as the chemically modified polymer were characterized by Fourier transform infrared (FTIR). The FTIR spectra obtained confirm that these are indeed the expected polymers. The results of the theoretical study are in good agreement with those obtained experimentally, and make it possible not only to predict the type of chemical modification reaction of this polymer, which is a nucleophilic substitution, but also to determine its order (SN<sub>2</sub>).

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**Conflicts of Interest:** The authors declare no conflict of interest.

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