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Synthesis and Characterization of Di-n-hexylcyclopentane A.G.Gasanov¹, S.T. Aliyeva¹, A.G.Azizov², A.M.Mammadova¹, S.R. Khalilova¹, I.G.Ayyubov¹

¹ Institute of Petrochemical Processes of Azerbaijan National Science Academy, Az 1025, Baku, Azerbaijan
²Institute of Polymer Materials of Azerbaijan NSA, Az 5004 Sumgait, Azerbaijan

Abstract

The synthesis of di-n-hexylcyclopentane has been based on alkylation reaction of cyclopentadiene with n-hexyl alcohol. So, after alkylation process, further hydrogenation of received intermediate di-n-hexylcyclopentadiene has carried out and di-n-hexylcyclopentane synthesized. The physico-chemical properties of obtained compound have studied and its application as a lubricant has determined.

Keywords: *lubricants*, *alkylation of cyclopentadiene*, *n-hexyl alcohol*, *viscosity*, *lubricating properties*

1. Introduction

It is known that, alkyl- and polyalkyl-cyclopentanes have good viscosity-flow properties which lead to the novel preconditions for its application as lubricant oils [1-4]. In this direction, the key compounds for the synthesis of them are alkyl- and poly-cyclopentadienes, which have wide application, as well [5].

In present work, the synthesis of di-n-hexylcyclopentane was carried out as two steps process which consisting of alkylation of the cyclopentadiene and further hydrogenation of obtained di-n-cyclopentadiene.

2. Experimental part

Cyclopentadiene was taken as a initial compound and it has the following physico-chemical properties: density $-0.976~\text{g/sm}^3$; $n_d^{20}-1.5050$; boiling point -170^{0}C (with destruction). While, the properties of n-hexyl alcohol is: density $-0.819~\text{g/sm}^3$, $n_d^{20}-1.4130$, boiling point -157.2^{0}C .

The IR-spectra of initial and synthesized compounds were registered at the spectrophotometer UR-20 in the field 700-4000 sm⁻¹. The NMR-spectra were taken on Bruker WP-400 (400 MHz). The chemical shifts were determined in comparison of TMS (when solvent is CDCl₃).

The synthesis of di-n-hexylcyclopentane was carried out by following method. 86 g of n-hexyl alcohol (2.65 mole) and 10.3 g of 87 % solution of KOH (0.16 mole) were poured into the reaction flask, provided with mechanical stirrer, dropped funnel, Din-Stark cap with cooler and thermometer. The portion of DCPD (6.6 g, 0.05 mole) were added to reaction mixture before heating. Then temperature was increased to 150°C. After water was gathered in Din-Stark cap, last portion of DCPD was added drop-by-drop to reaction mixture (28,4 g; 0,22 mole) during 1,5 h. After finishing it, reaction mixture was



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heated during for 4 h. After cooling, mixture was washed by water, hexane, then dried over MgSO₄ and distillated in vacuum (p=10 mm Hg). The physico-chemical properties of di-n-hexylcyclopentadiene was determined: boiling point $-72\text{-}75^{\circ}\text{C}$ (10 mm.Hg), density -0.7855 g/ml, $n_{d}^{20}-1.4340$.

The hydrogenation of obtained dinhexylcyclopentadiene was carried out at rolling autoclave in the present of Ni/Cr₂O₃ catalyst at temperature 200-220 $^{\circ}$ C, hydrogen pressure 30 atm. The following physico-chemical properties were determined for obtained new product: boiling point - 135 $^{\circ}$ C (at 10 mm. Hg.) $n_{\rm d}^{20} - 1,4475$, density – 0,8730 g/ml, viscosity index – 169, freezing point – 55 $^{\circ}$ C.

3. Result and Discussion

The alkylation reaction of cyclopentadiene with nhexyl alcohol and synthesis of di-nhexylcyclopentadiene was conducted by following scheme:

Where $R = n.C_6 H_{13}$;

Di-n-hexylcyclopentane was obtained by hydrogenation reaction of di-n-hexylcyclopentadiene in the presence of nickel-chrome catalyst.

The analysis of physico-chemical properties of received product shows that, the presence of low freezing point and high viscosity index creates the good premises to the application of this compound as lubricants.

In the IR-spectra of di-n-hexylcyclopentane, the adsoption stripes were founded in the fields 723, 890, 1054, 1119, 1375, 1450 sm⁻¹, which is fitting to valent and deformation waves of C-H in the methyl and methylene groups. Moreover, the adsorption stripe was founded also in the 2857-2922 sm⁻¹ field which is matching to double C=C bond.

The NMR¹H -spectra of di-n-hexylcyclopentane is described in Figure 1. As it is seen, the signals of methyl and methylene protons were found in the fields of 0,862 m.p. and 1,234-1,490 m.p., respectively. Moreover, the signal of cyclic methylene group is matching to the 2, 290 m.p. field. The signals of protons which place near double C=C bond are adsorbed at 3,527-3,805 m.p. field.

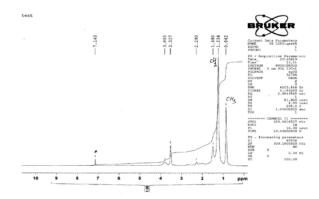


Fig.1. NMR¹H -spectra of di- n-hexylcyclopentane



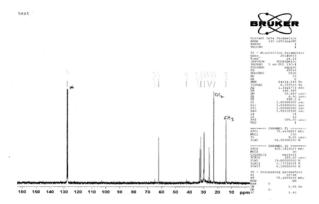


Fig.2. NMR¹³C-spectra of di-n-hexylcyclopentane

As it is shown in Figure 2, in the NMR¹³C-spectra of obtained di-n-hexylcyclopentane, the signals of carbon atoms were founded in the 13, 22, 25, 30-32 m.p. fields and it corresponds to carbon atoms from methyl, methylene, methyn and cyclic methylene groups, respectively. Moreover, 61 m.p. stripe responds to carbon atom of double bond.

4. Conclusions

Summarizing all, it is determined that, the method based on the alkylation of cyclopentadiene with n-hexyl alcohol in the presence of alkaline catalyst and further hydrogenation of obtained dinhexylcyclopentadiene is very useful for the synthesis of di-n-hexylcyclopentane. Additionally, the obtained di-n-hexylcyclopentane product has wide application as lubricant oils.

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Arif Hasanov is head of "Cycloolefins" laboratory at the Institute of Petrochemical Processes of Azerbaijan NAS. He is author of more than 230 scientific works including 7 books, 22 copywrite certificate, patents in Russian and Azerbaijan.