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SYNTHESIS OF *para*-[1(3)-METHYLCYCLOALKYL]PHENOLS: PROPERTIES AND APPLICATIONS

Ulviyya GURBANOVA, Chingiz RASULOV, Günay HEYDARLI, Fatima GASIMOVA

Academician Y.H. Mammadaliyev Institute of Petrochemical Processes of the Ministry of Science and Education of the Republic of Azerbaijan, AZ 1025, Baku, 30 Khojaly Ave
E-mail:ulviyyagurbanova01@gmail.com

ARTICLE INFO	ABSTRACT
<i>Article history</i>	<i>This research focuses on the cycloalkylation reactions of phenol with 1-methylcyclopentene (MCP) and 1(3)-methylcyclohexenes (MCH) were investigated using a zeolite-based heterogeneous catalyst of both ecological and industrial significance. The reactions were carried out in a continuous-flow pilot plant, and optimal reaction conditions were determined as follows: temperature – 120°C, phenol-to-MCH molar ratio – 1:1, and volumetric flow rate – 0.6 h⁻¹. Under these conditions, the yield of <i>para</i>-(1-methylcyclohexyl)phenol was found to be 73.8% with a selectivity of 92.1%; the yield of <i>para</i>-(3-methylcyclohexyl)phenol was 69.4% with a selectivity of 89.7%; and the yield of <i>para</i>-(1-methylcyclopentyl)phenol reached 68.5% with the same selectivity of 89.2%. The structure and composition of the synthesized products were confirmed by IR spectroscopy (BRUKER ALPHA) and by ¹H and ¹³C NMR spectroscopy (300 MHz). In addition, differential thermal analysis (DTA) and thermogravimetric analysis (TGA) were carried out using a Jupiter STA 449F3 synchronous thermal analyzer (NETZSCH, Germany) under dynamic conditions in an inert nitrogen atmosphere, within the temperature range of 25–650°C and at a heating rate of 10 K/min. Furthermore, the acylation reactions of the synthesized <i>para</i>-methylcycloalkylphenols with acetyl chloride in the presence of a ZnCl₂/γ-Al₂O₃ catalyst were studied.</i>
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1. Introduction

The rapid development of industry leads to an increased demand for chemical compounds and the expansion of their application areas. Simultaneously, pressing global issues such as environmental protection and energy efficiency are gaining increased attention. In this context, the synthesis of chemically efficient and environmentally safe compounds has become a priority in modern research. Among various classes of organic compounds, alkylphenols occupy a prominent position due to their broad applicability and valuable properties [1–5].

Alkylphenols not only serve as corrosion inhibitors, antioxidants, and stabilizers but also act as key components in the production of synthetic lubricants, surfactants, polymer additives, cosmetic products, and pharmaceuticals. The structural diversity and chemical reactivity of these compounds enable their widespread application in petrochemistry, polymer science, and materials engineering [6–10]. In addition to enhancing the thermal and mechanical stability of

end products, alkylphenols contribute to resistance against oxidative degradation, thereby improving the overall efficiency of industrial processes[11–15].

Growing environmental concerns have intensified research into developing more flexible and efficient synthesis methods for alkylphenols, as well as improving their functional characteristics. Consequently, the relevance of these compounds in chemical research and industrial applications has grown significantly. In light of these factors, the present study investigates the synthesis and application potential of industrially significant alkylphenols—namely, 1-methylcyclopentyl- and 1(3)-methylcyclohexylphenols—using an environmentally benign, zeolite-based catalyst KN-30 (containing 96.22% SiO_2), TS 2177-011-07622236-2008, under continuous-flow conditions in a pilot plant. The use of zeolites as catalysts in petrochemical processes is considered one of the major achievements of twentieth-century chemical science. Their large surface area, microporous structure, and intrinsic acidity make zeolites highly efficient heterogeneous catalysts. These properties not only facilitate faster reaction rates but also enhance product yield. Consequently, zeolite-based heterogeneous catalysts are widely employed in oil refining and petrochemical industries [16–20].

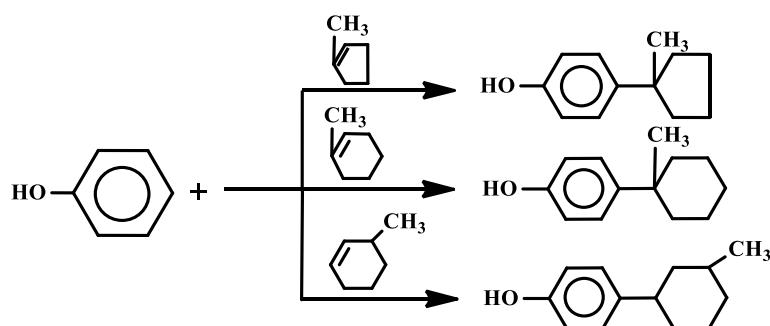
2. Experimental Section

Phenol, 1-methylcyclopentene, and 1(3)-methylcyclohexenes were used as starting materials for the alkylation process. Phenol was distilled to ensure purity before the reaction. 1-Methylcyclopentene was obtained via the dehydration of cyclohexanol. 1-Methylcyclohexene (with a purity of 99.8%) was synthesized through the condensation of isoprene with ethylene, while 3-methylcyclohexene (98.6% purity) was produced by the condensation of perylene with ethylene. The physical properties of the initial compounds are presented in the table below.

Table 1. Physical properties of the raw materials

Properties	Phenol	1-MCP	1-MCH	3-MCH
Chemical formula	$\text{C}_6\text{H}_5\text{OH}$	C_6H_{10}	C_7H_{12}	C_7H_{12}
Molecular weight (g/mol)	94	82	96	96
Melting point (°C)	40-42	-	-	-
Boiling point (°C)	182	75	110-111	103-104
Density (g/cm ³ , 25°C)	1.071	0.7782	0.8200	0.8142
Refractive index (n_D^{20} , 20°C)	1.4503	1.4347	1.4502	1.4435

The mechanism of the cycloalkylation reaction of phenol with 1-methylcyclopentene and 1(3)-methylcyclohexenes is based on the principles of electrophilic aromatic substitution (EAS). The chemical equation of the reaction is presented below.



The schematic illustration of the continuous-flow pilot plant used for the cycloalkylation process is presented below.

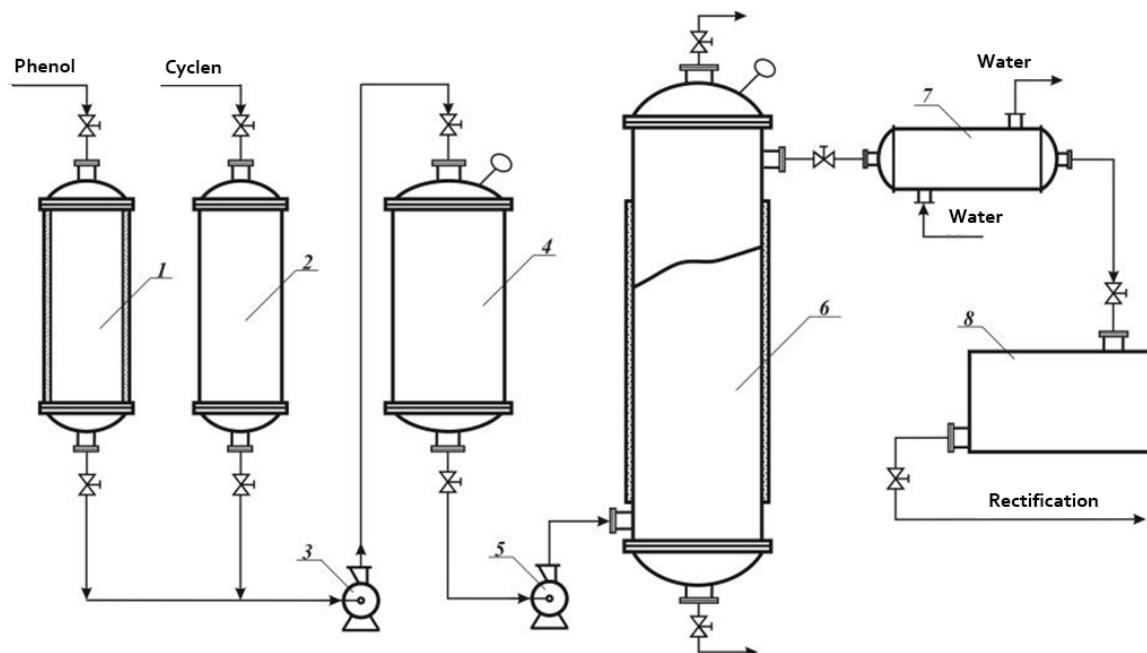


Fig. 1 Schematic representation of the technological process for the catalytic cycloalkylation of phenol with 1(3)-methylcycloalkenes

1 – Tank for phenol, 2 – Tank for cyclene, 3, 5 – Pumps, 4 – Mixer, 6 – Reactor, 7 -Condenser, 8 –Collection tank for alkylate

The phenol from tank (1) and the cyclene from tank (2) are fed into the mixer (4) at a predetermined ratio. The temperature in the phenol tank (1) is maintained at 40-45°C. The resulting mixture of phenol and cyclene is then directed from the mixer to the lower section of the reactor (6). The mixture of components passes over the catalyst and, after cooling in the condenser (7), is collected in the alkylate tank (8), from which it is directed to the rectification unit. During the rectification process, unreacted methylcyclohexene is first separated, followed by phenol and the target products under vacuum conditions (10 mm Hg). Once the experiment is completed, the raw material feed is halted, and the remaining product in the reactor is transferred to the receiving tank. After 500 hours of operation, the reactor is then switched to the regeneration mode.

The continuous operation of the phenol alkylation process in a pilot-scale plant provides a significant foundation for its potential industrial application. Conducting the process in such systems not only helps reduce production costs but also facilitates the development of a simpler and more practical technological scheme.

As an example, Figure 2 presents the results of the cycloalkylation reaction of phenol with 1-methylcyclohexene.

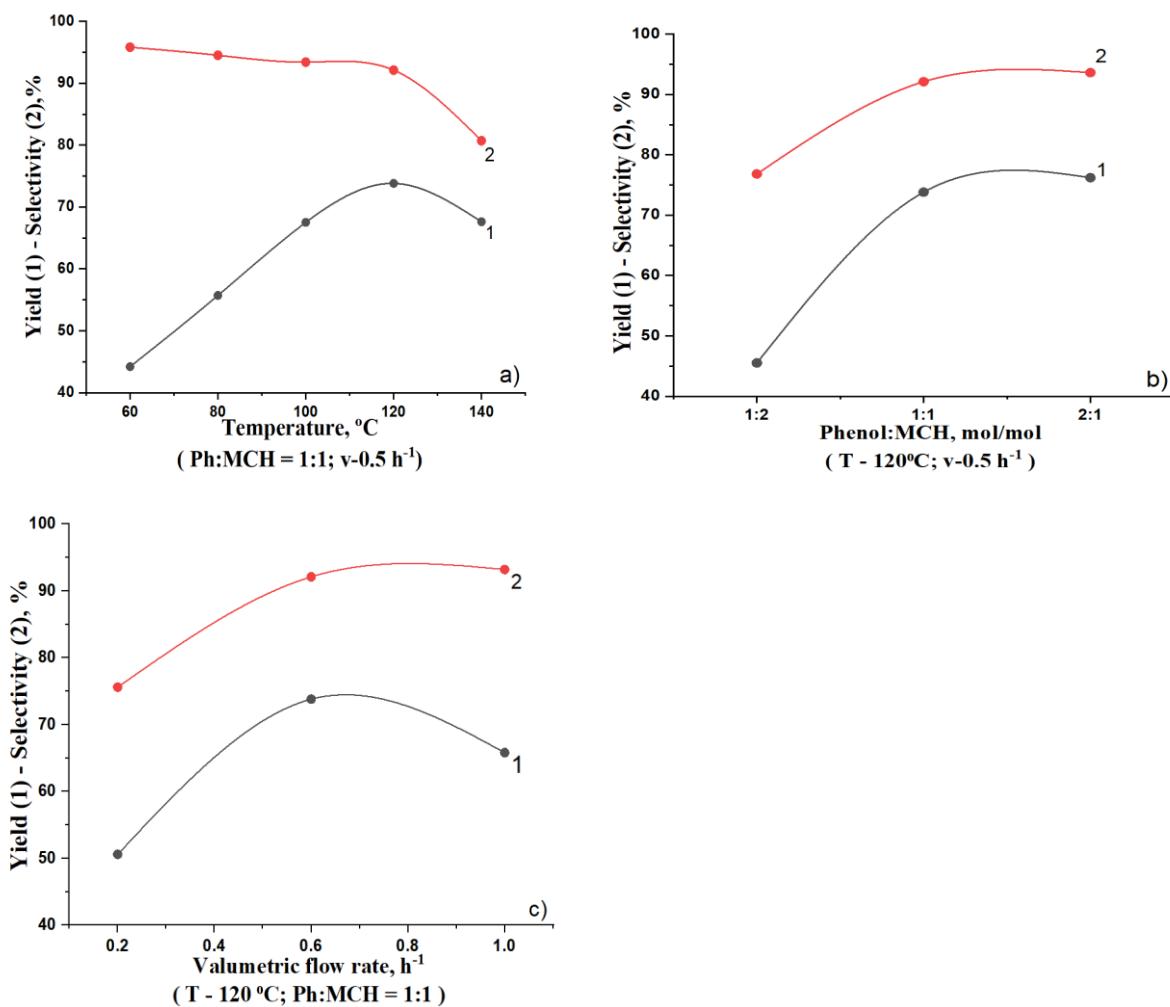
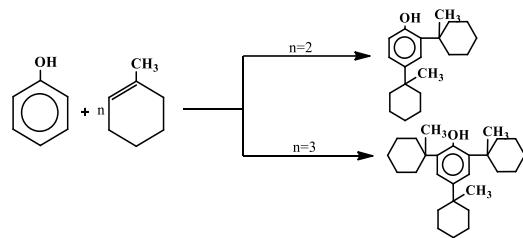


Fig. 2 Dependence curves of the yield (1) and selectivity (2) of *para*-(1-methylcyclohexyl)phenol on temperature (a), molar ratios of phenol to 1-MCH (b), and volumetric flow rate (c)

The study on the synthesis of *para*-(1-methylcyclohexyl)phenols examined the effects of temperature, the molar ratio of phenol to 1-MCH, and volumetric flow rate on the yield and selectivity of the target product. The reaction temperature for *para*-(1-methylcycloalkyl)phenols was investigated within the range of 60–140 °C, the phenol-to-MCH molar ratio varied from 1:2 to 2:1 (mol/mol), and the volumetric flow rate was studied between 0.2 and 1.0 h⁻¹.

As shown in Figure 2, increasing the temperature from 60 to 120 °C results in an increase in the yield of the target product from 44.2 to 73.8% (calculated based on phenol conversion). Within this temperature range, selectivity remains nearly constant, varying between 93.4 and 96.2%. However, when the temperature reaches 140 °C, the yield decreases to 67.6%, and selectivity drops to 80.7%. This decline is attributed to the formation of undesirable isomers at elevated temperatures.

The amount of MCH in the reaction mixture also plays a crucial role. Under conditions of excess MCH, the yield of the target product decreases to 45.5%. This is attributed to the formation of di- and tri-substituted methylcyclohexylphenols.



When the molar ratio of phenol to methylcyclohexene (MCH) is 1:1, the yield of *para*-(1-methylcyclohexyl)phenol reaches 73.8 with a selectivity of 92.1%. Increasing the amount of phenol in the reaction mixture results in a slight improvement in yield and selectivity, reaching 76.2 and 93.6%, respectively. However, this marginal increase is not considered economically significant, as the additional consumption of phenol does not lead to a substantial advantage in productivity or selectivity.

When the volumetric flow rate was investigated within the range of 0.6–1.0 h⁻¹, it was found that at a flow rate of 0.6 h⁻¹, the yield of the target product and the selectivity of the process were satisfactory (Fig. 2).

Thus, the optimal conditions for the continuous cycloalkylation of phenol with 1-methylcyclohexene over the KN-30 catalyst in a pilot-scale setup were established as follows: temperature of 120°C, phenol-to-1-MCH molar ratio of 1:1, and a volumetric flow rate of 0.6 h⁻¹. Under these optimal conditions, the yield of *para*-(1-methylcyclohexyl)phenol (based on phenol conversion) reached 73.8%, with a selectivity (based on the target product) of 92.1%.

Similarly, the continuous cycloalkylation reactions of phenol with 1-methylcyclopentene and 3-methylcyclohexene were investigated over the KN-30 catalyst. The cycloalkylation process was carried out under the optimal conditions previously established for the reactions of phenol with 1-MTP and 1-MCH. Under these conditions, the yield of *para*-(1-methylcyclopentyl)phenol based on phenol conversion reached 68.5%, with a selectivity of 89.2% relative to the target product. The yield and selectivity of *para*-(3-methylcyclohexyl)phenol were 69.4 and 89.7%, respectively.

3. Results and discussion

The figures below present the analysis results of *para*-(1-methylcyclopentyl)- and *para*-(1-methylcyclohexyl)phenols obtained by IR, ¹H and ¹³C NMR, differential thermal analysis (DTA), and thermogravimetric (TG) methods.

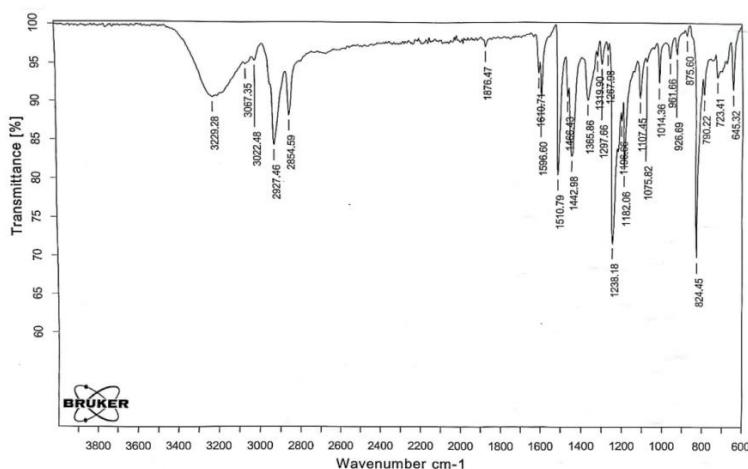


Fig. 3 IR Spectrum of *para*-(1-methylcyclopentyl)phenol

The IR spectrum of *para*-(1-methylcyclopentyl)phenol exhibited the following absorption bands:

- 824 cm⁻¹ – *para*-substituted benzene ring;
- 926, 961 cm⁻¹ – C–H bonds of the cyclic ring;
- 1014, 1075, 1107, 1182, 1238 cm⁻¹ – stretching vibrations of the phenol C–O bond in the C–OH group;
- 1365, 1442, 1466 cm⁻¹ – deformation vibrations of C–H bonds in CH₃ and CH₂ groups;
- 1510, 1596, 1610 cm⁻¹ – stretching vibrations of the C=C bonds in the HC=CH group;
- 2854, 2927 cm⁻¹ – C–H stretching vibrations in CH₃ and CH₂ groups;
- 3022, 3067 cm⁻¹ – stretching vibrations of the O–H bond in the CH=CH group;
- 3229 cm⁻¹ – stretching vibration of the O–H bond in the COH group.

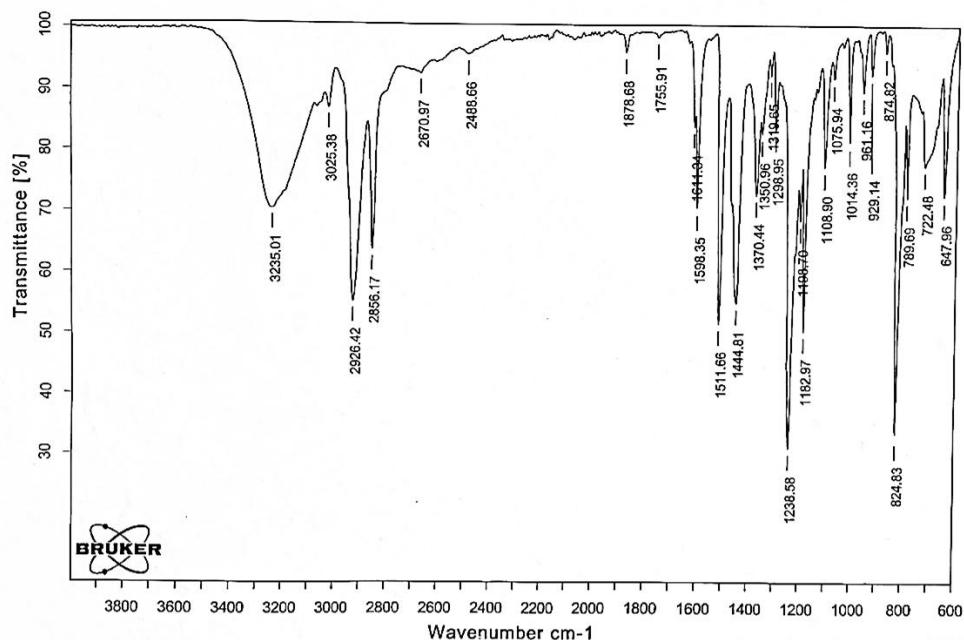


Fig 4. IR Spectrum of *para*-(1-methylcyclopentyl)phenol

In the IR spectrum of *para*-(1-methylcyclohexyl)phenol, the following characteristic absorption bands were observed: 824 cm⁻¹ – indicative of a *para*-substituted benzene ring; 647, 722, 789 cm⁻¹ – out-of-plane and in-plane deformation vibrations of C–H bonds; 929, 961 cm⁻¹ – deformation vibrations of the cyclohexyl ring; 1014, 1075, 1182, 1196, 1238 cm⁻¹ – stretching vibrations of the C–O bond; 1365, 1442, 1466 cm⁻¹ – deformation vibrations of C–H bonds in CH₃ and CH₂ groups; 2854, 2927 cm⁻¹ – stretching vibrations of C–H bonds in CH₃ and CH₂ groups; 1510, 1596, 1610 cm⁻¹ – stretching vibrations of C=C bonds in the aromatic ring (-HC=CH- group); 3022, 3067 cm⁻¹ – stretching vibrations of aromatic C–H bonds; 3229 cm⁻¹ – stretching vibration of the O–H bond in the phenolic group.

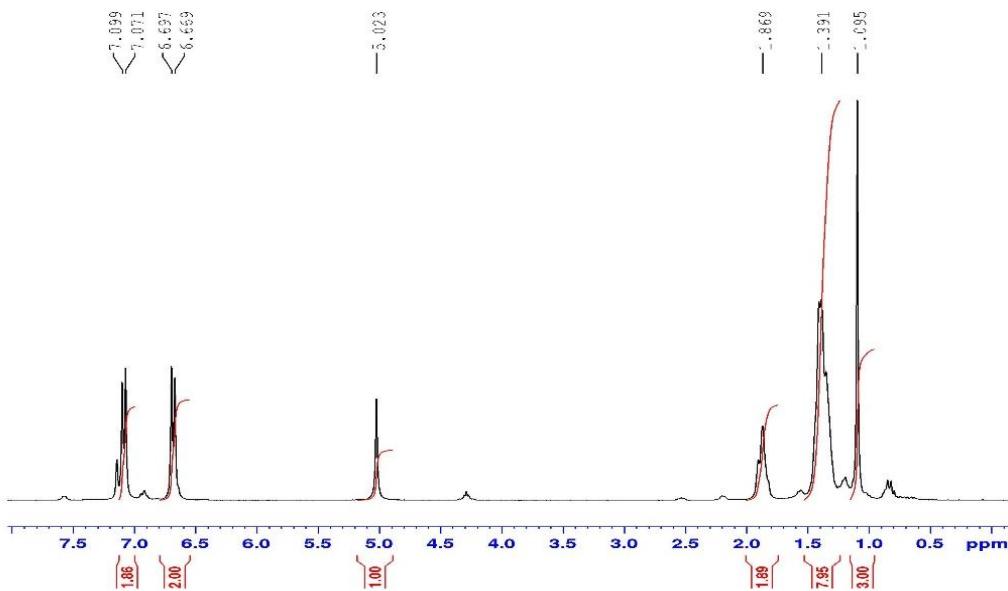


Fig 5. ^1H NMR spectra of *para*-(1-methylcyclopentyl)phenol

The results of the ^1H NMR spectroscopic analysis of *para*-(1-methylcyclopentyl)phenol (CDCl_3 , δ , ppm) are as follows:

- 1.09–1.85 (multiplet, 8H, 4 CH_2 groups of the cyclopentyl ring);
- 1.37 (singlet, 3H, CH_3 methyl group on the cyclopentyl ring);
- 4.70 (singlet, 1H, OH group);
- 6.64 (doublet, 1H, aromatic proton);
- 7.08 (doublet, 1H, aromatic proton);
- 7.13 (singlet, 1H, aromatic proton).

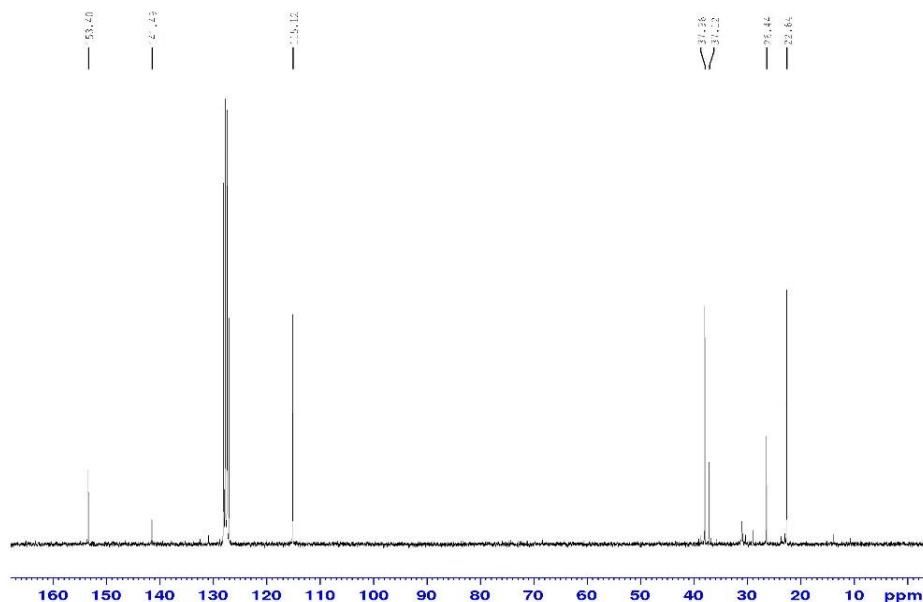


Fig 6. ^{13}C NMR spectra of *para*-(1-methylcyclopentyl)phenol

The results of the ^{13}C NMR spectroscopic analysis of *para*-(1-methylcyclopentyl)phenol (CDCl_3 , δ , ppm) are as follows:

- 22.6 (CH_3 , methyl group on the cyclopentyl ring);
- 26.8, 28.3, 34.5 (CH_2 groups, cyclopentyl ring);
- 43.1 (CH , quaternary carbon connected to the methyl group on the cyclopentyl ring);
- 115.2, 124.4, 129.6 (aromatic carbons);
- 138.9 (C–C, substituted carbon in the aromatic ring);
- 152.2 (C–OH, carbon bonded to the hydroxyl group in the phenol ring).

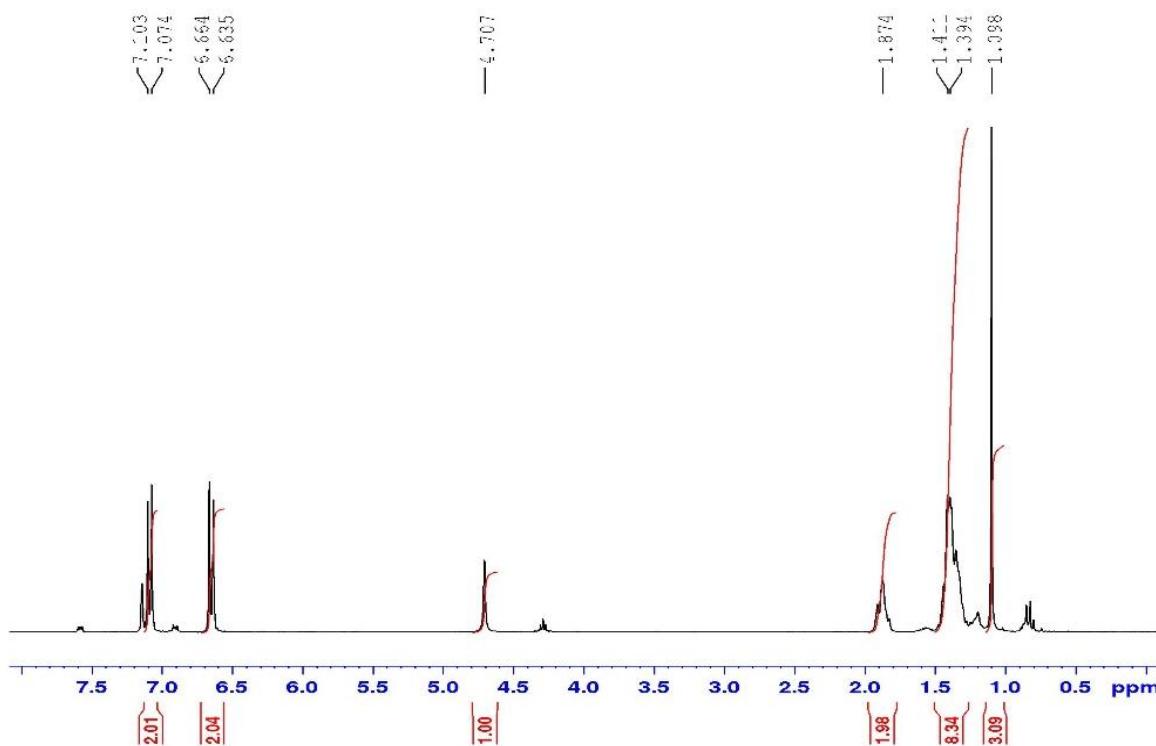


Fig. 7 ^1H NMR spectra of *para*-(1-methylcyclohexyl)phenol

The results of the ^1H NMR spectroscopic analysis for *para*-(1-methylcyclohexyl)phenol (in CDCl_3 , δ , ppm) are as follows: 1.09–1.87 ppm (multiplet, 10H): corresponding to five CH_2 groups in the cyclohexyl ring; 1.39 ppm (singlet, 3H): methyl group attached to the cyclohexyl ring; 4.70 ppm (singlet, 1H): phenolic OH group; 6.63–6.67 ppm (doublet, 1H): aromatic proton; 7.07–7.10 ppm (doublet, 1H): aromatic proton; 7.10 ppm (singlet, 1H): aromatic proton

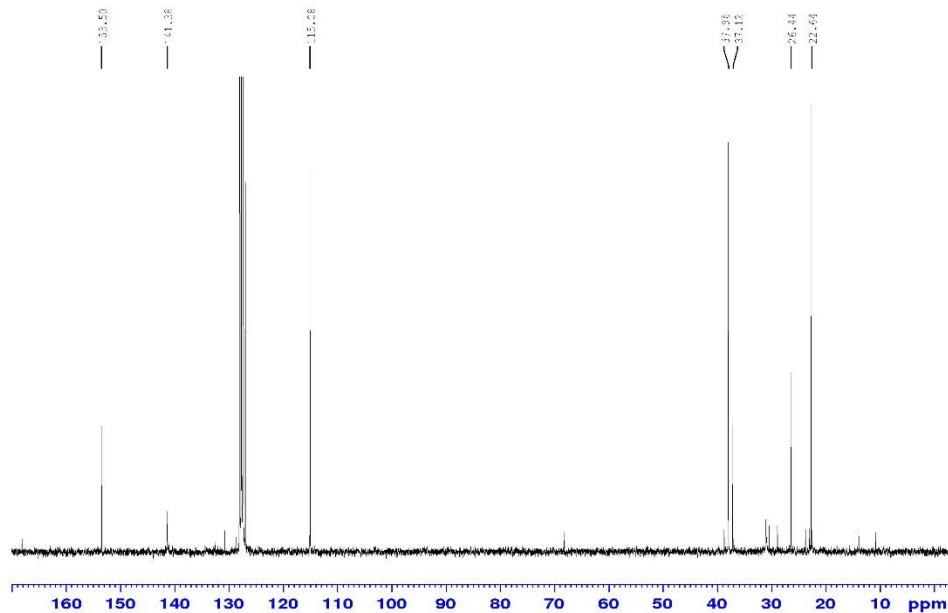


Fig 8. ^{13}C NMR spectra of *para*-(1-methylcyclohexyl)phenol

The results of the ^{13}C NMR spectroscopic analysis for *para*-(1-methylcyclohexyl)phenol: Aromatic carbons: 153.5, 141.4, 139.5, 131.4, 129.3, 115.0 ppm; cyclohexyl ring carbons: 37.9, 37.1 ppm – quaternary carbons of the cyclohexyl ring; 26.4 ppm – CH_2 groups in the cyclohexyl ring; 22.6 ppm – CH group in the cyclohexyl ring; 31.0 ppm – methyl group attached to the cyclohexyl ring

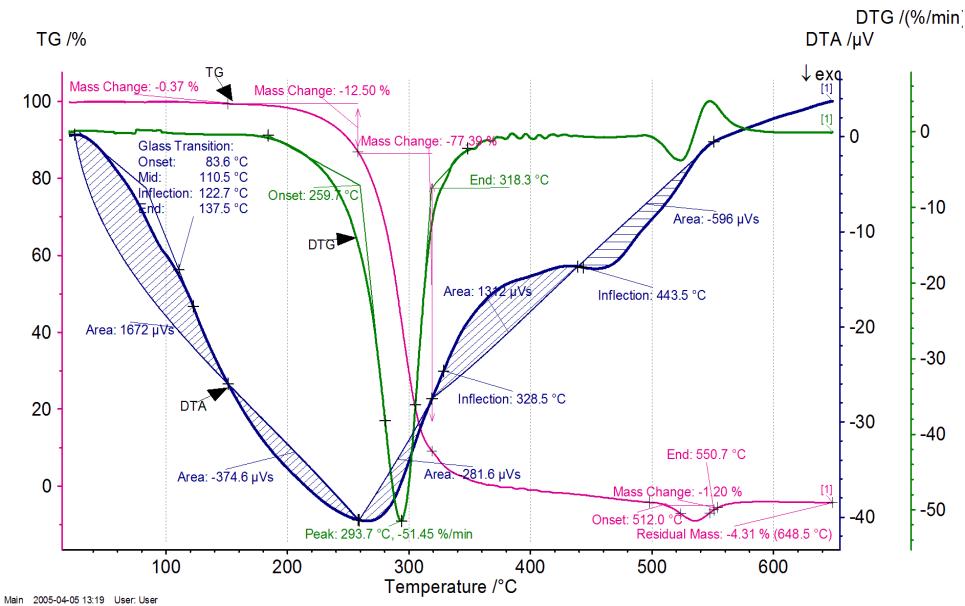


Fig. 9 TG/DTA thermogram of *para*-(1-methylcyclopentyl)phenol

Thermal analysis results of *para*-(1-methylcyclopentyl)phenol indicate that the compound undergoes a glass transition phase between 83.6°C and 137.5°C, characterized by increased molecular mobility within its amorphous structure without any observed mass loss. The main degradation occurs within the temperature range of 259.0°C to 318.3°C, during which a mass loss of 12.50% is recorded. The maximum decomposition rate is observed at 293.7°C, reaching -51.45%/min. Subsequently, a second major degradation takes place between 318.3°C and

550.7°C, with a mass loss of 77.39%. An exothermic process is observed in the final stage, leaving a residual mass of 4.31% at the end of the analysis. Overall, the compound can be considered thermally stable up to 250°C.

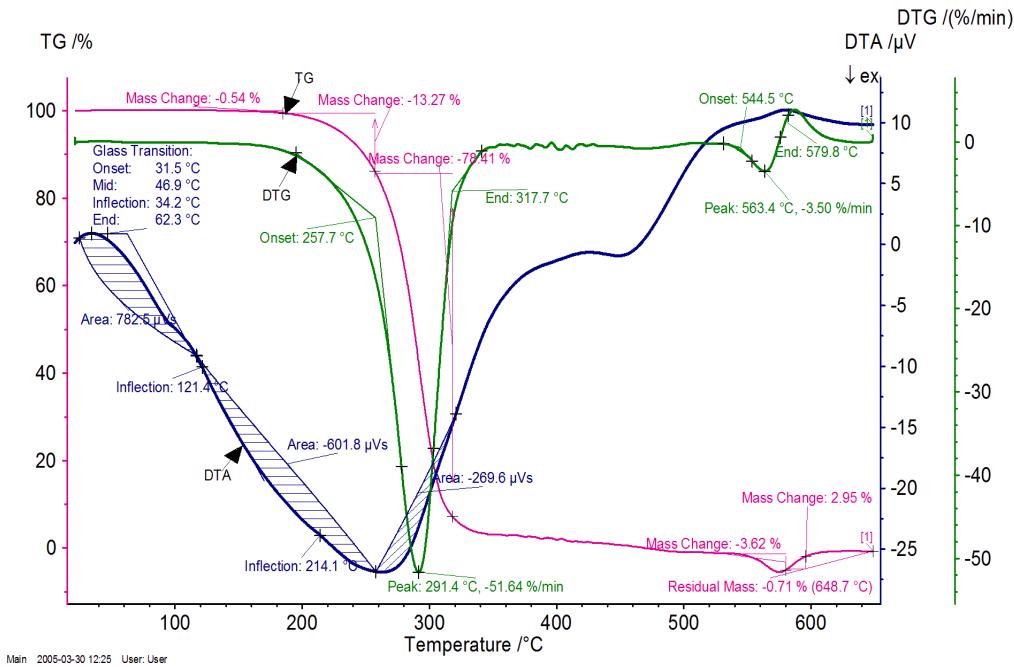


Fig. 10 TG/DTA thermogram of *para*-(1-methylcyclohexyl)phenol

According to the thermogravimetric analysis results, *para*-(1-methylcyclohexyl)phenol experiences an initial mass loss of approximately 0.54% within the temperature range of 31.5°C to 62.3°C. This loss is primarily attributed to the evaporation of adsorbed water and other volatile components on the compound's surface. The main thermal degradation occurs between 257.7°C and 317.7°C, with a recorded mass loss of 78.41%, which corresponds to the breakdown of the compound's primary carbon skeleton. The maximum decomposition rate is observed at 291.4°C, reaching -51.64%/min, indicating rapid degradation at this temperature. Based on these findings, *para*-(1-methylcyclohexyl)phenol can be considered thermally stable up to approximately 250°C, making it suitable for applications under conditions below this temperature.

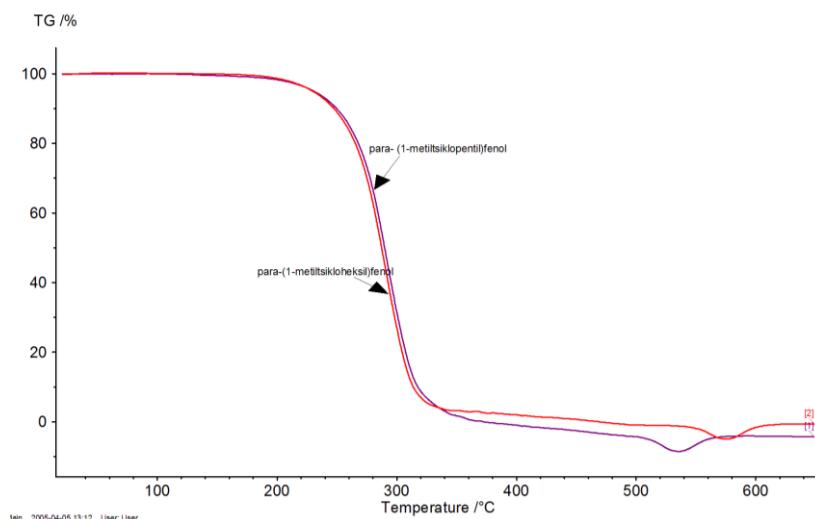
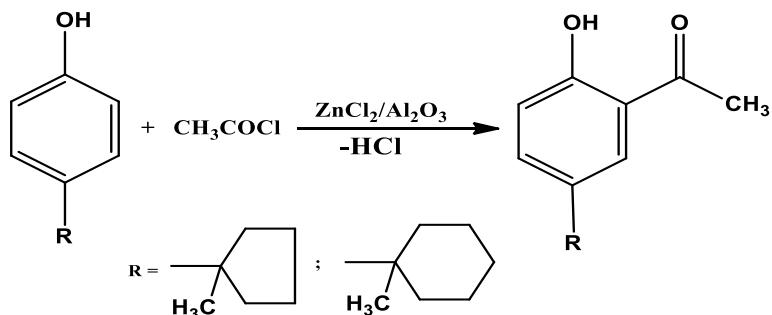


Fig. 11 Combined TG curve of *para*-(1-methylcyclopentyl)phenol and *para*-(1-methylcyclohexyl)phenol

Table 2. The table presents the physicochemical characteristics and elemental composition of *para*-(1-methylcycloalkyl)-phenols

Structural formula	Temp., °C/10 mm Hg	Temp., °C	Mol. Mass	Elemental composition, %	
				calculated	found
				C	H
	145-148	90	176	81.8 81.3	9.1 8.3
	161-164	96	190	82.1 81.5	9.5 8.6
	158-169	91	190	82.1 81.2	9.5 8.4

In the next stage, the obtained *para*-(1-methylcyclopentyl)- and *para*-[1(3)-methylcyclohexyl]phenols were subjected to acylation reactions in the presence of a dispersed $ZnCl_2$ -supported γ - Al_2O_3 catalyst. The acylation was carried out at temperatures ranging from 120 to 160°C for durations of 20 to 60 minutes, with molar ratios of cycloalkylphenol to acetyl chloride varying from 1:0.5 to 1:3. The reactions were conducted using 20 wt% $ZnCl_2/\gamma$ - Al_2O_3 catalyst.



The acylation reaction of *para*-methylcycloalkylphenols with acetyl chloride was performed in a three-necked laboratory flask equipped with a thermometer, stirrer, and dropping funnel, using a $ZnCl_2/\gamma$ - Al_2O_3 catalyst. The study investigated the effects of various factors such as temperature, reaction time, and molar ratio of reagents on the yield and selectivity of the target products. Optimal reaction conditions were determined as follows: at 140°C, for 30 minutes, with a molar ratio of cycloalkylphenol to acetyl chloride of 1:2 (mol/mol), the yields of the desired products ranged from 63.5% to 67.4%. The obtained 2-hydroxy-5-(1-methylcyclopentyl)- and 2-hydroxy-5-[1(3)-methylcyclohexyl]acetophenones were evaluated as photostabilizers for polystyrene, antioxidants in M-8 motor oil, and antirad additives in polypropylene. The results were positive.

para-[1(3)-Methylcycloalkyl]phenols obtained from the alkylation of phenol are widely used as intermediates in the petrochemical industry. Methylcyclohexylphenols undergo aminomethylation reactions with amines to form Mannich bases, which are utilized in various applications including as photo- and thermostabilizers in polymer materials, surfactants in lubricants, additives in rubber products, and pesticides widely used in agriculture — for example, as fungicides against powdery mildew in viticulture, insecticides against the Colorado potato beetle

in potato farming, as well as bactericides and disinfectants for diseases affecting both large and small livestock. These compounds have extensive applications in various other fields as well.

4. Conclusion

1. Cycloalkylation reactions of phenol with 1-methylcyclopentene and 1(3)-methylcyclohexenes were investigated in a continuous-flow laboratory setup using the industrial zeolite-based KN-30 catalyst. The optimal reaction conditions were established as follows: temperature of 120°C, molar ratio of phenol to 1(3)-methylcyclohexene of 1:1, and a volumetric flow rate of 0.6 h⁻¹. Under these conditions, the yields of *para*-[1(3)-methylcyclohexyl]phenols ranged from 69.4 to 73.8% with selectivities between 89.7 and 92.1%. The yield and selectivity of *para*-(1-methylcyclopentyl)phenol were 68.5 and 89.2%, respectively.
2. For the acylation of *para*-[1(3)-methylcycloalkyl]phenols using dispersed ZnCl₂-supported γ -Al₂O₃ catalyst, optimal reaction conditions were determined as follows: temperature of 140°C, reaction time of 30 minutes, and a cycloalkylphenol to acetyl chloride molar ratio of 1:2. The yields of the synthesized target products ranged from 63.5 to 67.4%.

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