

## Highly efficient copper–palladium nanoalloy catalysts on modified carbon supports

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Catalytic systems were made using Cu, Pd, and Pd-Cu nanoparticles (NPs) supported on modified activated carbon (ACm) of BAU-A grade, which had been modified with hydrochloric acid, for dehydrochlorination of chlorobenzene and 1,2-dichlorobenzene. Based on previous studies, the optimal content of metal NPs in the heterogeneous catalysts for the transformation of mono- and dichlorobenzene organochlorine pollutants utilizing the method of catalytic dehydrochlorination were determined to be: 5% for Pd; 10% for Cu; and, 3% Pd and 7% Cu for the bimetallic variant. The metal NPs were determined to bond to the carboxyl and carbonyl functional groups of the ACm. The characteristics of the catalysts were studied using FTIR spectroscopy, differential thermogravimetric analysis, scanning electron microscopy and adsorption porosimetry. Finally, following dehydrochlorination, a chromatograph mass spectrometer was used to identify the products. These results on the pore structures of the catalysts demonstrate good development, allowing for an increased number of sites to adsorb persistent organic pollutants (POPs). Whereas all the catalysts showed effectiveness in dehydrochlorination of chlorobenzene and 1,2-dichlorobenzene, separately, into benzene, the bimetallic catalyst, 3Pd-7Cu/ACm, demonstrates the best results, with conversion rates of 93.94% and 89.79%, respectively.

**Keywords:** hydrodechlorination, copper nanoparticles, palladium nanoparticles (NPs), catalysis, bimetallic catalyst, organochlorine compounds, modified carbon, persistent organic pollutants (POPs).

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### 1. Introduction

Halogen-containing organic compounds are extensively utilized in various industries and by consumers, serving as coolants, medicines and their delivery systems, solvents, and plasticizers, and many other applications. Due to their high toxicity and chemical stability, numerous halogenated organic compounds are identified as persistent organic pollutants (POPs). Consequently, many of these compounds are banned

by the World Health Organization (WHO). On May 22, 2001, the Stockholm Convention on Persistent Organic Pollutants was signed. This agreement aims to reduce and eventually eliminate existing stocks of POPs, as well as to cease the production, use, and release of any new POPs [1]. The currently accumulated reserves of excessively produced halogen-containing by-products require environmentally safe handling. This necessitates the development of new methods for their conversion and disposal. Modern methods

of neutralizing waste containing POPs utilize reductive dechlorination. This process can be optimized to save more re-sources by using new types of nanocatalysts that regenerate the hydrocarbon component of halogenated molecules [2].

The development of reductive methods for the dechlorination of organohalogen compounds is facilitated by the search for active, selective, and efficient catalysts. Several studies have demonstrated that catalysts based on palladium (Pd) and platinum (Pt) exhibit the highest activity and selectivity in the reductive transformation of chlorine-containing compounds [3-7]. This is attributed to their capacity to facilitate the separation of  $H_2$ , thereby promoting the cleavage of the C–Cl bond [8]. Many studies have been devoted to the analysis of catalysts based on Pd and Pt for the electrocatalytic reduction of halogenated, specifically chlorine-containing, organic compounds. Typically, the content of Pd and Pt in such catalysts reaches 10% by weight [9-12]. However, the utilization of noble metals increases the cost of catalysts and thus, the entire process. In recent years, there has been a trend to alleviate the cost of catalysts by diluting or substituting these expensive noble metals with others that are more economical yet still exhibit acceptable characteristics.

Carbon carriers are highly promising for catalytic applications due to their purity, exceptional porous structure, and high specific surface area [13]. Activated carbon (AC), in particular, is extensively utilized as a carrier material in the synthesis of catalysts aimed at recycling persistent organic pollutants (POPs) [14-17]. Granular activated carbon (GAC) combined with bimetallic Pd and Fe nanoparticles (NPs) was used for the simultaneous adsorption and dehalogenation of polychlorinated biphenyl (PCB) [17]. For this purpose, the pore volume of granular activated carbon is impregnated with a solution of Fe (III) nitrate  $Fe(NO_3)_3$ , followed by heat treatment at a temperature of 3000C. Subsequent reduction with sodium borohydride ( $NaBH_4$ ) leads to the formation of zerovalent (ZVI)  $Fe^0$ , with Fe NPs sizes in the range of 7-40 nm. Afterwards, the reduced Pd solution ( $Pd(CH_3CO_2)_2$ ) is added to the surface of Fe NPs. As a result, small stable Fe NPs of 6-12 nm in size are formed in the mesopores of the granular AC, on which a 2-3 nm Pd nanolayer has been uniformly distributed. The GAC/ZVI/Pd system demonstrated 90% efficiency in the dechlorination of 2-chlorobiphenyl to form the reaction product biphenyl. In previous studies, the hydrodechlorination of organohalogen substances with Pd catalysts supported on coal-based material was 80-100% [18-22]. Some re-

searchers developed na-nosized palladium catalysts for the reductive dechlorination of PCBs, achieving this at low temperatures and with a minimal amount of catalyst [23-26]. Fifteen PCB congeners, including monochlorinated (PCB 1, PCB 2), dichlorinated (PCB 4, PCB 5, PCB 7, PCB 9, PCB 10, PCB 11, PCB 12, PCB 14, PCB 15), trichlorinated (PCB 29), tetrachlorinated (PCB 77), pentachlorinated (PCB 126), and hexachlorinated (PCB 169), under-went complete decomposition. This process resulted in a 100% yield of biphenyl using 10% Pd/C–Et<sub>3</sub>N as a catalyst [27]. In this case, the reductive dechlorination of PCBs was carried out at room temperature in MeOH, with bottled hydrogen as a reducing agent, Pd (10%)/C, and Et<sub>3</sub>N. 2.5 mg of catalyst was consumed per 25 mg of PCB.

Despite the high catalytic activity of palladium, the high cost of noble metals limits the large-scale production of these catalysts. Recently, copper nanoparticles (Cu NPs) have garnered significant interest in the development of new catalytic systems due to their high activity and selectivity [28, 33-35]. A study on copper catalysts, with metal contents ranging from 0.5% to 5.0% (wt.), demonstrated the significant impact of the carrier material on alachlor conversion, achieving conversion rates ranging from 85.2% to 92.9% [34]. A catalytic system utilizing copper nanoparticles (Cu NPs) stabilized with polyvinylpyrrolidone (PVPD40) and deposited onto an activated carbon (AC) substrate achieved a chlorobenzene conversion rate of 94.46% [14].

Considering that Cu catalysts for hydrodechlorination have very often been used in conjunction with noble metals, the high conversion rate of alachlor hydrodechlorination is an interesting result from the point of view of scaling [29-32].

Compared to previously reported Pd–Cu catalytic systems, the novelty of this work lies in the use of a Cu-rich composition, which makes it possible to significantly reduce the amount of expensive noble metal without loss of catalytic efficiency. Additionally, the application of HCl-modified wood-based activated carbon as a support is an important distinguishing factor, since chemical modification increases the number of oxygen-containing functional groups and improves the interaction of the metal nanoparticles with the surface, which can promote the formation of more homogeneous and stable Pd–Cu nanoalloys. These two factors together – optimized Pd/Cu ratios and targeted chemical modification of the carbon carrier – create conditions for enhanced Pd–Cu synergy and demonstrate that high hydrodechlorination efficiency can be achieved not only by increasing

Pd loading, but also by engineering the support and nanoalloy composition.

This study focuses on the synthesis and investigation of the physical and chemical properties of Cu, Pd, and Pd-Cu catalysts to evaluate their effectiveness in the liquid-phase hydrodechlorination of mono- and dichlorobenzenes.

## 2. Materials and Methods

### 2.1. Materials

In this study, the following reagents were used: copper (II) nitrate  $\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$  (99.9%), palladium(II) chloride  $\text{PdCl}_2$  (99.9%), sodium tetrahydroborate  $\text{NaBH}_4$  (99.9%), sodium hydroxide  $\text{NaOH}$ , chlorobenzene  $\text{C}_6\text{H}_5\text{Cl}$  (99.5%), and 1,2-dichlorobenzene  $\text{C}_6\text{H}_4\text{Cl}_2$  (99%). All reagents were purchased from Sigma Aldrich and used without additional purification. As a catalyst support, commercial wood-based activated carbon BAU-A (GOST 6217-74, Russia) was employed, which possessed the following characteristics: iodine adsorption activity of not less than 60%, ash content not exceeding 6%, and moisture content not exceeding 6%. For the modification of activated carbon, hydrochloric acid (GOST 11125-84, LLCMK MAGNA, Russia) was used according to the procedure described by Shaimardan et al [13].

### 2.2. Methods

#### 2.2.1 Preparing the catalysts

In this study, the catalysts were synthesized using the wet impregnation method described by Hy-

eok et al. (Fig. 1) [32–34]. For the preparation of the Cu catalyst, 6.5 g of copper(II) nitrate was dissolved in 5 mL of deionized water, after which 10 g of  $\text{AC}_m$  was introduced into the solution and stirred vigorously for 10 minutes. The mixture was then placed in a desiccator for 4 hours to facilitate the sorption of Cu ions, followed by drying at 150 °C for 2 hours to remove excess water. To stabilize the Cu ions, the  $\text{Cu}^{2+}/\text{AC}_m$  sample was calcined at 330 °C for 4 hours. Subsequently, the reduction of  $\text{Cu}^{2+}$  in  $\text{AC}_m$  was carried out by dissolving 1.6 g of sodium borohydride ( $\text{NaBH}_4$ ) in 20 mL of distilled water. Subsequently, the  $\text{NaBH}_4$  solution was added to a 50 mL aqueous-alcohol solution with a volume ratio of 30:70, and the mixture was stirred. To adjust the pH to  $\leq 7$ , a 5 N  $\text{NaOH}$  solution was introduced. The  $\text{NaBH}_4$  solution was slowly added to the  $\text{Cu}^{2+}/\text{AC}_m$  suspension and stirred for 3 hours until the complete release of hydrogen. The resulting catalyst contained 10 wt.% of active Cu phase (10Cu/ $\text{AC}_m$ ).

The preparation of the Pd catalyst followed a similar procedure, where 20 mg of palladium (II) chloride was dissolved in 20 mL of an aqueous-alcoholic solution with a volume ratio of 30:70 (90% ethanol to water), and then mixed with 1 g of  $\text{AC}_m$  under vigorous stirring. The obtained catalyst contained 5 wt.% of active Pd phase (5Pd/ $\text{AC}_m$ ). The bimetallic Pd–Cu catalyst was synthesized using the same procedure, with the difference that  $\text{Pd}^{2+}/\text{AC}_m$  and  $\text{Cu}^{2+}/\text{AC}_m$  suspensions were combined in a 3:7 ratio before the addition of the  $\text{NaBH}_4$  solution. The final bimetallic catalyst contained 3 wt.% of active Pd phase and 7 wt.% of active Cu phase (3Pd–7Cu/ $\text{AC}_m$ ).

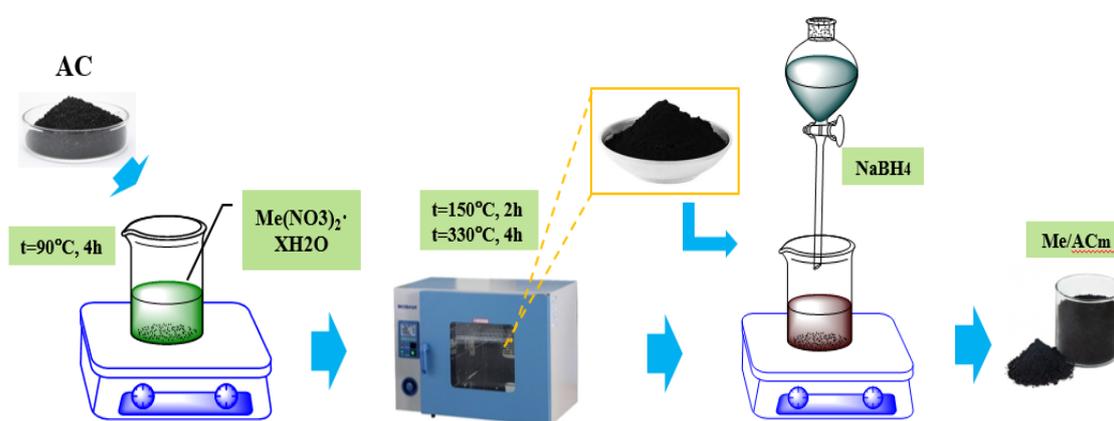


Figure 1 – Schematic for preparation of 10Cu/ $\text{AC}_m$ , 5Pd/ $\text{AC}_m$  and 3Pd-7Cu/ $\text{AC}_m$  catalysts.---

### 2.2.2 FTIR analysis

Fourier-transform infrared (FTIR) spectroscopy was performed using an FTIR FT-801 spectrometer (Simex, Russia) with a resolution of  $1\text{ cm}^{-1}$  in the range of  $500\text{--}4000\text{ cm}^{-1}$ . Measurements were conducted at  $25^\circ\text{C}$  following the standard procedure, employing KBr pellets prepared at a 1:10 sample-to-KBr ratio. A total of 100 scans were collected for each spectrum. Prior to use, potassium bromide was finely ground and calcined at  $200^\circ\text{C}$  for 3 hours.

### 2.2.3 X-ray diffraction (XRD) analysis

The crystal structure of the samples was analyzed using an X'PertPRO diffractometer (Malvern Panalytical Empyrean, Netherlands) equipped with monochromatized  $\text{CuK}\alpha$  radiation. Data were collected over the  $2\theta$  range of  $10\text{--}45^\circ$  with a step size of  $0.02^\circ$ . The operating conditions included an X-ray tube voltage of 45 kV, a current of 30 mA, and a counting time of 0.5 s per step.

### 2.2.4 Thermogravimetric analysis (TGA)

Thermal stability and decomposition behavior of the catalysts were investigated using an STA449C simultaneous thermal analyzer (NETZSCH, Germany) under an argon atmosphere. Measurements were conducted in the temperature range of  $30\text{--}700^\circ\text{C}$  with a heating rate of  $10 \pm 1^\circ\text{C}/\text{min}$ . The initial sample mass was approximately  $20 \pm 2\text{ mg}$ .

### 2.2.5 Scanning and transmission electron microscopy (SEM and TEM)

The morphology and structure of the catalysts were characterized using a Crossbeam 540 high-vacuum scanning electron microscope (Zeiss, Germany). Elemental composition was determined by energy-dispersive X-ray spectroscopy (EDS, Thermo Fisher Scientific, USA). Transmission electron microscopy (TEM) images were obtained with a JEM-1400 microscope (JEOL, Japan), operating at an accelerating voltage of 120 kV, with a resolution of 0.38 nm, equipped with a Morada high-resolution CCD digital camera (Olympus, Japan). Prior to TEM, samples were ground in an agate mortar, dispersed in ethanol, and sonicated at 44 kHz using an UZDN-2T ultrasonic generator (Electron, Russia). A drop of the suspension was deposited on a perforated carbon-coated copper grid and dried before imaging. Particle size distributions and average particle diameters were determined statistically from TEM micrographs.

### 2.2.6 Adsorption porosimetry

Textural properties of the catalysts were determined by low-temperature nitrogen adsorption-desorption measurements using an Autosorb-1 analyzer (Quantachrome Instruments, USA). Before analysis, the samples were degassed under vacuum at  $200\text{--}250^\circ\text{C}$  for 3 hours. The Brunauer-Emmett-Teller (BET) method was applied to calculate specific surface area, with a measurement error of  $\pm 2.8\%$ . Pore size distribution and total pore volume were derived from desorption isotherms using the Barrett-Joyner-Halenda (BJH) method.

### 2.2.7 Catalytic activity

Catalytic hydrodechlorination of chlorobenzene and 1,2-dichlorobenzene was carried out in an RVD-2-150 high-pressure reactor (Uoslab, Ukraine). The reaction mixture consisted of 1 mL of organochlorine compound, 0.1 g of catalyst, 3 mL of NaOH solution, and 1 mL of ethanol in a two-neck round-bottom flask [30]. Hydrogen gas was introduced at  $50^\circ\text{C}$  under 10 atm pressure, with magnetic stirring for 5 hours. After completion, the reaction mixture was washed with 10 mL of magnesium sulfate solution to precipitate sodium salts. A 1 mL aliquot of the treated solution was diluted with 19 mL of hexane for subsequent analysis.

### 2.2.8 Gas chromatography-mass spectrometry (GC-MS)

The hydrodechlorination products were analyzed using a 5975C GC/MS system (Agilent, USA) equipped with a GC-MSD quadrupole mass spectrometric detector (Agilent, USA). Operating conditions were as follows: electron ionization energy of 70 eV; HP-5MS quartz capillary column ( $30\text{ m} \times 0.25\text{ mm} \times 0.25\text{ }\mu\text{m}$ ); helium as carrier gas; split ratio of 1:50; flow rate of 1.0 mL/min. The oven temperature program consisted of an initial temperature of  $40^\circ\text{C}$  (held for 3 min), followed by heating at  $10^\circ\text{C}/\text{min}$  to a maximum of  $290^\circ\text{C}$ , with a final holding time of 30 min.

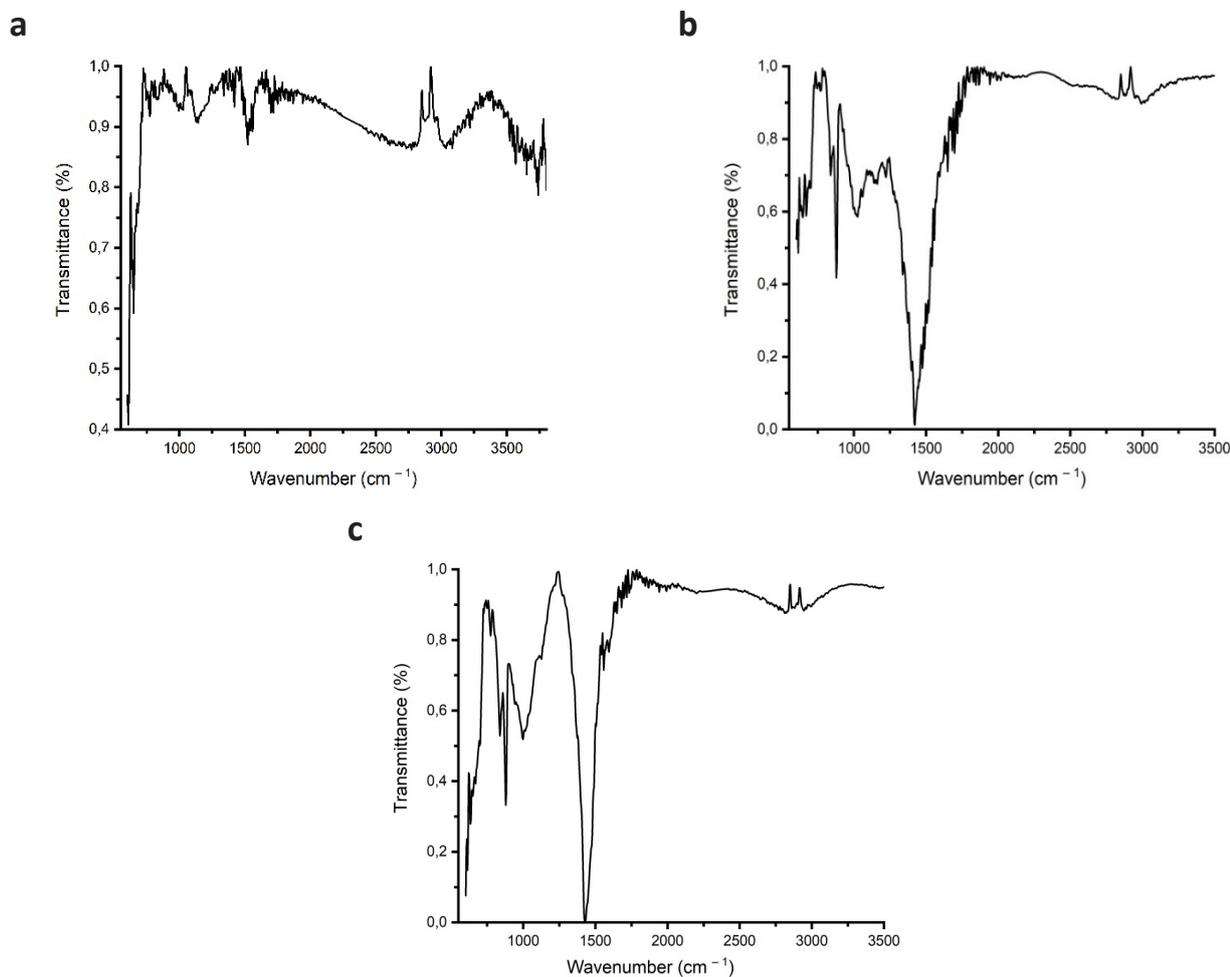
## 3. Results and discussion

Physical and chemical characteristics of the 5Pd/ACm, 10Cu/ACm, and 3Pd-7Cu/ACm catalysts are presented below.

### 3.1. FTIR spectroscopy

In the FTIR spectra of 5Pd/AC<sub>m</sub>, 10Cu/AC<sub>m</sub> and 3Pd-7Cu/AC<sub>m</sub>, in comparison to unmodified AC<sub>m</sub>, the appearance of intense peaks in the region of 600-750 cm<sup>-1</sup> and the absence of an absorption band in the region of 1713 cm<sup>-1</sup>, related to C=O, are observed, which

indicates a new bond in the catalysts, as seen in Figure 2. The data is in good agreement with previous studies. The most important difference in the FTIR spectra of the catalytic systems is the absence of the C=O vibration at 1713 cm<sup>-1</sup>, which indicates the interaction of NPs and AC<sub>m</sub> through the C–OH functional group.



**Figure 2** – The FTIR spectra of catalytic systems: a) 5Pd/AC<sub>m</sub>; b) 10Cu/AC<sub>m</sub>; and, c) 3Pd-7Cu/AC<sub>m</sub>.

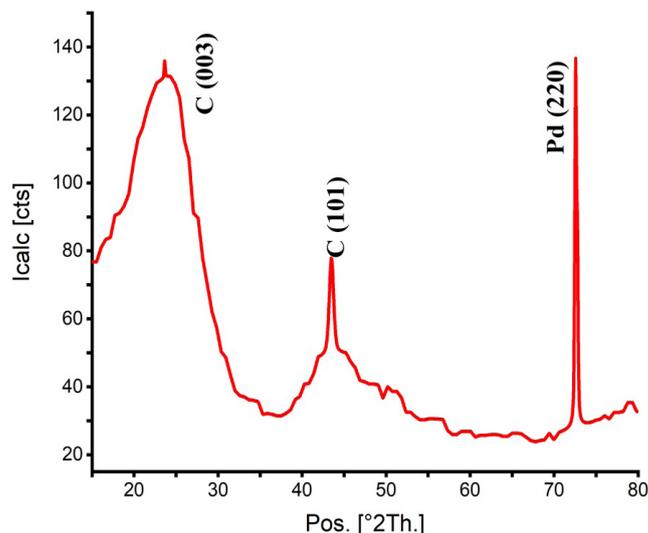
FTIR spectra demonstrate the disappearance of the C=O vibration at 1713 cm<sup>-1</sup> together with the appearance of new bands in the 600–750 cm<sup>-1</sup> region, which evidences chemical coordination of Pd and Cu nanoparticles specifically with carbonyl / carboxyl surface functionalities on AC<sub>m</sub>. XRD further confirms that the metals do not exist as independent separate phases: the presence of mixed reflections characteristic for Pd–Cu al-

loy formation shows that the two metals interact electronically at the atomic level, forming common crystallographic domains rather than isolated particles. Taken together, FTIR and XRD results indicate that the active phase is a true nanoalloy, strongly bonded to oxygen-containing anchoring sites of the HCl-modified carbon support, and this metal–metal–support coupling is the basis for the enhanced catalytic behavior observed.

### 3.2. XRD analysis

The X-ray diffraction (XRD) analysis of the 5Pd/AC<sub>m</sub> catalyst reveals three distinct diffraction peaks at  $2\theta$  values of 23.6°, 43.5°, and 72.5°, which correspond to the Miller indices (003), (101), and (220),

respectively. These peaks indicate that the catalyst exhibits a face-centered cubic lattice structure. This structural information is significant as it confirms the successful reduction of the PdCl<sub>2</sub> precursor to Pd NPs (Fig. 3, Table 1).



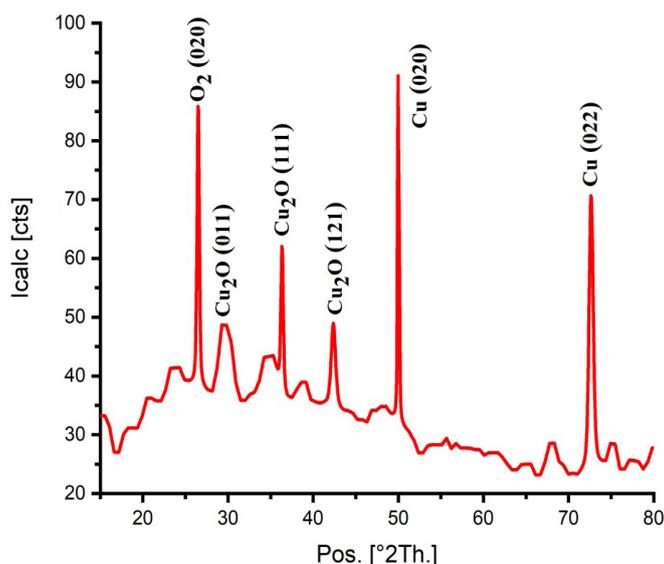
**Figure 3** – X-ray diffraction pattern of 5Pd/AC<sub>m</sub> catalyst.

**Table 1** – Interplanar distance (d) of atoms in the 5Pd/AC<sub>m</sub> catalyst.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
23.6308	4.90	0.0900	3.76197	4.52
43.4566	19.83	0.5904	2.08245	18.31
72.5528	108.30	0.1968	1.30296	100.00

XRD analysis of 10Cu/AC<sub>m</sub> revealed five diffraction peaks, with Miller indices at  $2\theta = 26.47$  (020),  $36.31$  (111),  $42.34$  (121),  $49.96$  (020) and  $72.58$  (022) (Fig. 4, Table 2). The X-ray diffraction (XRD) pattern of the 10Cu/AC<sub>m</sub> catalyst provides critical structural information. In the  $2\theta$  angle range of 50-80°, the pattern

exhibits a series of reflections characteristic of a face-centered cubic (FCC) lattice, thereby confirming the presence of metallic copper (Cu). Additionally, in the  $2\theta$  angle range of 20-45°, the pattern reveals reflections indicative of cubic lattices, which demonstrate the presence of copper oxide (CuO).



**Figure 4** – X-ray diffraction pattern of 10Cu/AC<sub>m</sub> catalyst.

**Table 2** – Interplanar distance (d) of atoms in the 10Cu/AC<sub>m</sub> catalyst.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
26.4737	34.14	0.2952	3.36688	61.29
36.3197	17.76	0.2952	2.47358	31.89
42.3402	9.88	0.5904	2.13474	17.74
49.9641	55.70	0.1476	1.82542	100.00
72.5885	35.69	0.4920	1.30241	64.08

The crystal structure and phase characteristics of the 3Pd-7Cu/AC<sub>m</sub> bimetallic catalyst are elucidated by five diffraction peaks, corresponding to the following Miller indices:  $2\theta = 26.49^\circ$  (002),  $36.88^\circ$  (100),  $43.14^\circ$  (111),  $62.51^\circ$  (213), and  $72.63^\circ$  (022) (Fig. 5, Table 3). The lattice parameter for palladium (Pd) is

determined to be  $3.36421 \text{ \AA}$ , which is closely aligned with the literature value of  $3.890 \text{ \AA}$ . The copper (Cu) component exhibits a face-centered cubic (FCC) lattice, while the palladium (Pd) displays a hexagonal lattice structure. Moreover, the Pd-Cu alloy demonstrates a tetragonal lattice structure (Table 3).

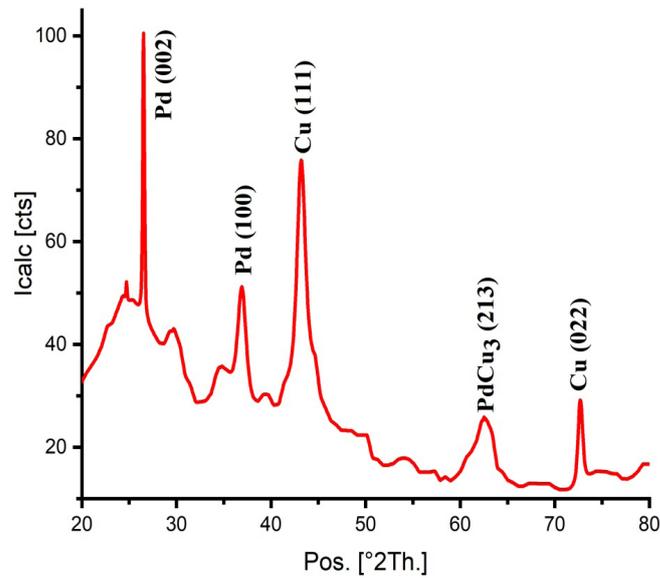


Figure 5 – X-ray diffraction of 3Pd-7Cu/AC<sub>m</sub>.

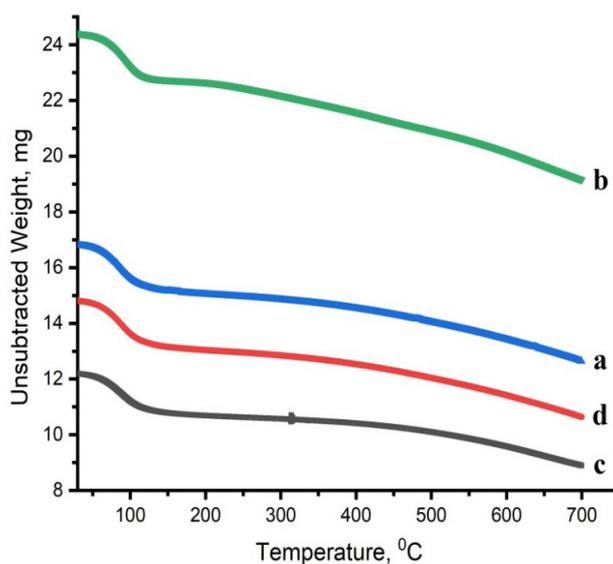
Table 3 – Interplanar distance (d) of atoms in the 3Pd-7Cu/AC<sub>m</sub> catalyst.

Pos. [°2Th.]	Height [cts]	FWHM Left [°2Th.]	d-spacing [Å]	Rel. Int. [%]
26.4950	40.78	0.1968	3.36421	100.00
36.8896	14.00	0.9840	2.43666	34.32
43.1491	28.14	0.9840	2.09658	69.01
62.5193	0.79	0.0900	1.48444	1.94
72.6397	12.30	0.4920	1.30161	30.16

### 3.3. Thermogravimetric analysis

Thermogravimetric analysis established a sharp decrease in the mass of all samples of catalysts, which occurred in the temperature range of 30-130°C, due to the evaporation of surface adsorbed water (Fig. 6). Furthermore, gradual decrease in the mass of 5Pd/AC<sub>m</sub>, 10Cu/AC<sub>m</sub> and 3Pd-7Cu/AC<sub>m</sub> was observed

in the temperature range of 450-700°C, which was caused by the combustion of carbonaceous compounds (Figures 5a-d). The state of general equilibrium in the temperature range of 150-450°C indicates the stability of the carboxyl, carbonyl and lactone groups, which is likely due to the interaction of metal ions with these specified functional groups.

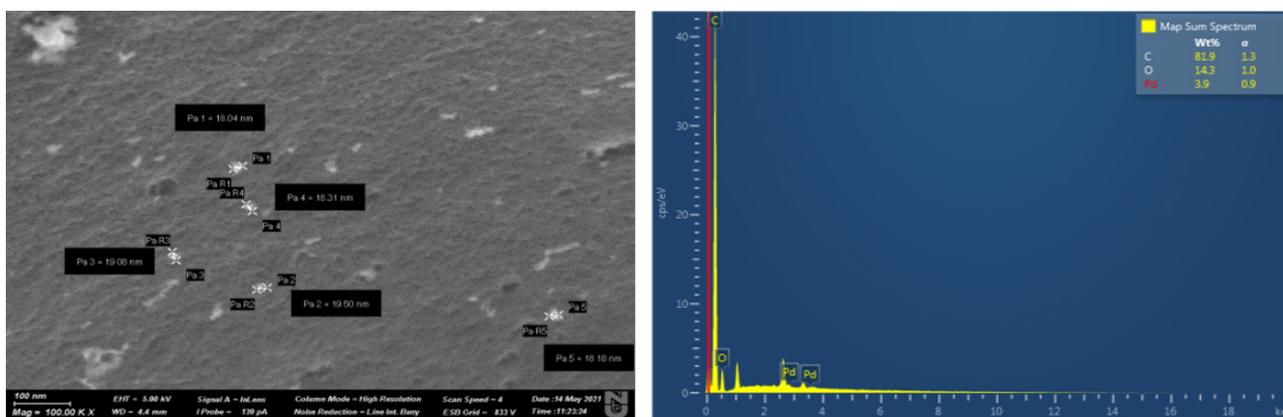


**Figure 6** –TGA curves of  $AC_m$  alone and of the catalytic systems: a)  $AC_m$ ; b)  $5Pd/AC_m$ ; c)  $10Cu/AC_m$ ; and, d)  $3Pd-7Cu/AC_m$ .

### 3.4. SEM and EDX analysis

Figures 6-8 present scanning electron micrographs of the  $5Pd/AC_m$ ,  $10Cu/AC_m$ , and  $3Pd-7Cu/AC_m$  catalysts. The  $5Pd/AC_m$  catalyst exhibits a relatively smooth surface texture, spherical palladium

nanoparticles (Pd NPs) with a uniform size distribution ranging from 18.04 to 19.50 nm (Fig. 7). Energy dispersive spectroscopy (EDX) analysis, integrated with SEM data, confirms that the palladium content in this catalyst is 3.9%.



**Figure 7** – SEM image and energy-dispersive X-ray (EDX) spectrum of the of  $5Pd/AC_m$  catalyst.

In the 10Cu/AC<sub>m</sub> catalyst, SEM imaging reveals a rougher surface texture with copper nanoparticles (Cu NPs) ranging in size from 18.58 to 29.56 nm

(Fig. 8). EDX data indicate a high carbon content of 79.9 wt.% and an oxygen content of 19.2 wt.%, while the copper content is measured at 10%

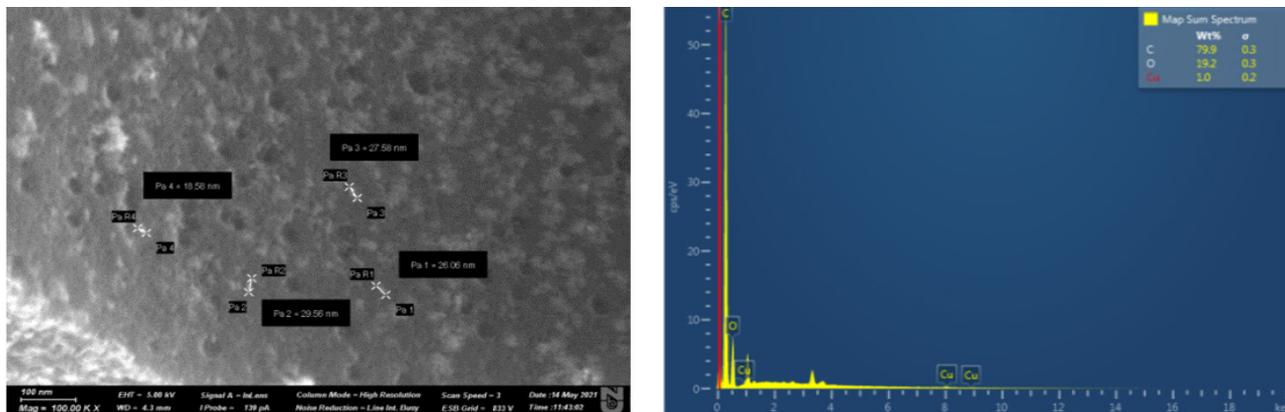


Figure 8 – SEM image and energy-dispersive X-ray (EDX) spectrum of the 10Cu/AC<sub>m</sub> catalyst

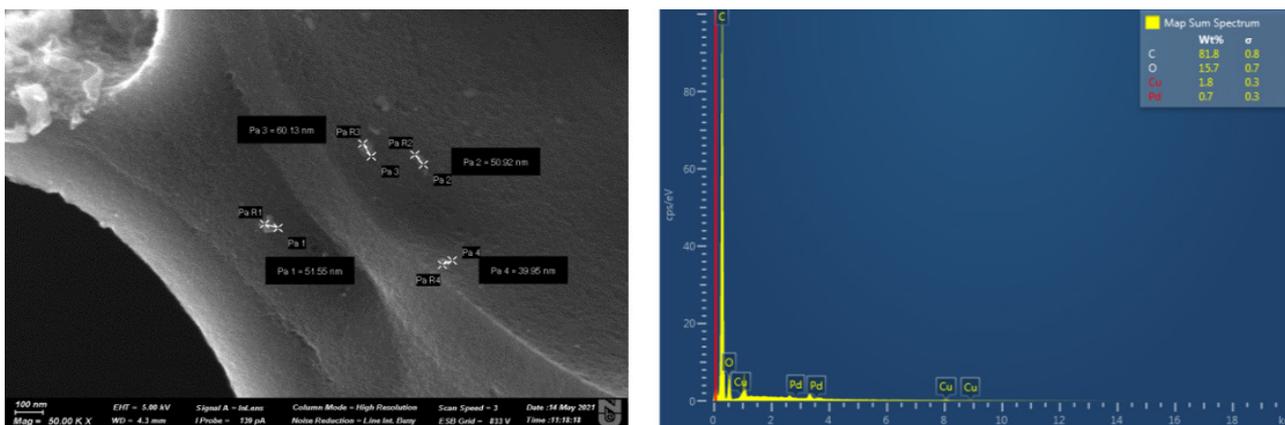


Figure 9 – SEM image and energy-dispersive X-ray (EDX) spectrum of the 3Pd-7Cu/AC<sub>m</sub> catalyst.

The 3Pd-7Cu/AC<sub>m</sub> bimetallic catalyst exhibits a more uniform texture compared to the monometallic catalysts, with particle sizes approximately twice as large, ranging from 39.95 to 60.13 nm (Fig. 9).

EDX analysis confirms the presence of copper (Cu) and palladium (Pd) at concentrations of 1.8 wt.% and 0.7%, respectively. Figures 10-12 depict the TEM images of 5Pd/AC<sub>m</sub>, 10Cu/AC<sub>m</sub>, and 3Pd-7Cu/AC<sub>m</sub>.

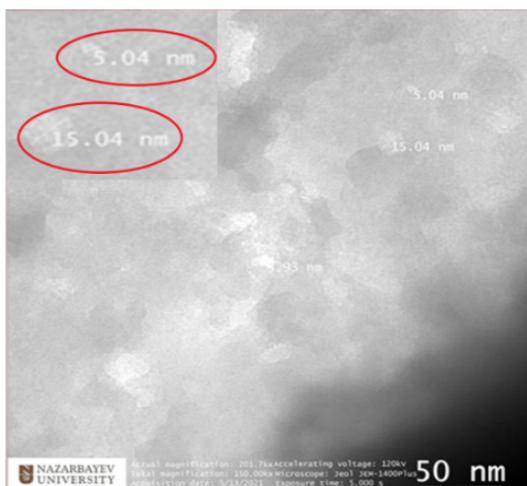


Figure 10 – TEM image of 5Pd/AC<sub>m</sub> catalyst.

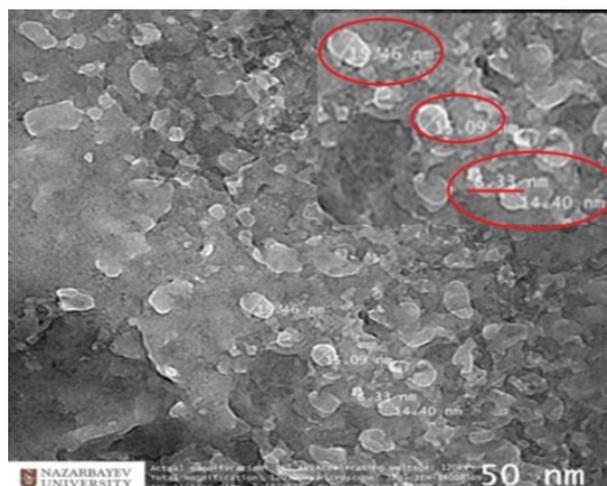


Figure 11 – TEM image of 10Cu/AC<sub>m</sub> catalyst.

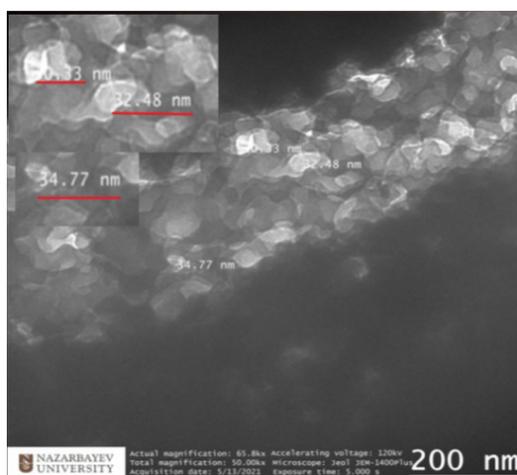


Figure 12 – TEM image of 3Pd-7Cu/AC<sub>m</sub> catalyst.

### 3.5. TEM analysis

Transmission electron microscopy reveals that Pd nanoparticles form on the surface layer (Fig. 10) and exhibit diverse geometries and dimensions, ranging from 5 to 15 nm. The 10Cu/AC<sub>m</sub> sample contains both small and large Cu nanoparticles, with sizes ranging from 8 to 19 nm, displaying various shapes and distributed uniformly across the entire surface of the catalyst (Fig. 11). The 3Pd-7Cu/AC<sub>m</sub> bimetallic catalyst features larger nanoparticles in the range of 30 to 34 nm, which aggregate to form agglomerates (Fig. 12).

### 3.6. BET analysis

Table 4 shows the indicators of the specific surface area of AC<sub>m</sub> alone and the three catalytic systems, calculated using the multipoint BET method,

including the total pore volume and characteristics of the micropores. Further, the pore size distribution, surface area, and nitrogen adsorption-desorption isotherms of the catalytic systems are shown in Figures 13-14.

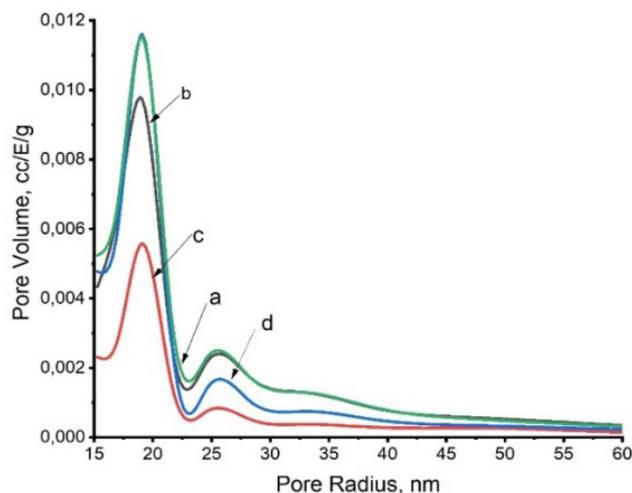
The catalysts synthesized exhibit a specific surface area ranging from 371.4 to 672.1 m<sup>2</sup>/g, which is 1.6 to 2.9 times higher than that of the AC<sub>m</sub> support (Table 4). Additionally, there is a notable reduction in pore size accompanied by a substantial increase in total pore volume. Upon incorporation of Pd nanoparticles into the modified carrier, the specific surface area reaches 371.4 m<sup>2</sup>/g. In contrast, catalysts containing Cu nanoparticles show a specific surface area twice as large as that of Pd, at approximately 672.1 m<sup>2</sup>/g. The 3Pd-7Cu/AC<sub>m</sub> catalytic system demonstrates a significant nearly two-fold increase in

specific pore surface area compared to the modified carrier (Table 4), consistent with findings from prior research [12]. All catalysts exhibit an increase in total pore volume, measured at 36.6, 73.9, and 65.6 ml/g for 5Pd/AC<sub>m</sub>, 10Cu/AC<sub>m</sub>, and 3Pd-7Cu/AC<sub>m</sub>, respectively, indicating a well-developed porous structure. This expansion suggests an enhanced capability for adsorbing pollutants due to an increase in available adsorption sites. The pore sizes (Dv(r)) of the catalysts is seen to have decreased by 6.37-6.4 times, as

compared to those of the AC<sub>m</sub> carrier [12]. The order of reduction is: 10Cu/AC<sub>m</sub> > 3Pd-7Cu/AC<sub>m</sub> > 5Pd/AC<sub>m</sub> (Table 4). The increase in the specific surface area and pore volume of all the catalysts is accompanied by a corresponding decrease in pore size, which is in the range of 19.084 nm, 19.168 nm and 19.070 nm for 5Pd/AC<sub>m</sub>, 10Cu/AC<sub>m</sub> and 3Pd-7Cu/AC<sub>m</sub>, respectively, as compared to the carrier [12] (Table 4). This result may be due to the localization of Pd, Cu and Pd-Cu inside the pores of AC<sub>m</sub>.

**Table 4** – Specific surface area, size, total pore volume of AC<sub>m</sub>; 5Pd/AC<sub>m</sub>; 10Cu/AC<sub>m</sub>; 3Pd-7Cu/AC<sub>m</sub>.

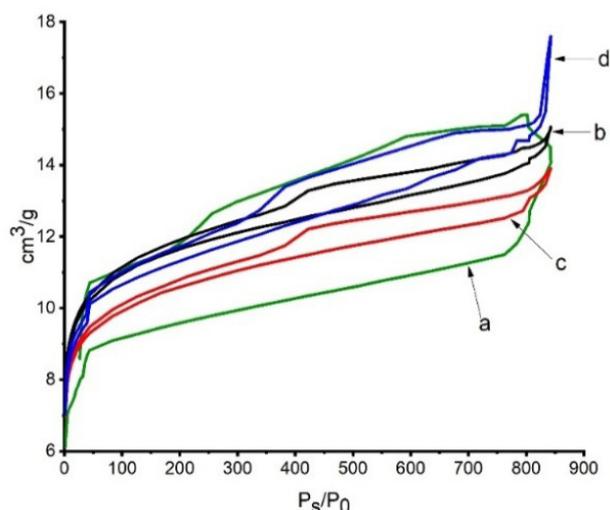
Sample	Surface area, m <sup>2</sup> /g	Poresize, nm	Porevolume, ml/g
AC <sub>m</sub> [12]	233.8	122.1	0.19
5Pd/AC <sub>m</sub>	371.4	19.0	36.6
10Cu/AC <sub>m</sub>	672.1	19.1	73.9
3Pd-7Cu/AC <sub>m</sub>	600.4	19.07	65.6



**Figure 13** – Pore size distribution of different catalysts: a) AC<sub>m</sub>; b) 5Pd/AC<sub>m</sub>; c) 10Cu/AC<sub>m</sub>; and, d) 3Pd-7Cu/AC<sub>m</sub>.

Further examination of the textural properties of the synthesized catalysts reveals distinct pore structures compared to the AC<sub>m</sub> carrier (Fig. 13). The N<sub>2</sub> adsorption isotherm for all catalysts exhibits a type IV behavior, characterized by rapid and uniform nitrogen adsorption. The initial curves of these isotherms indicate the presence of micropores

in all samples. At higher P/P<sub>0</sub> values, a slight capillary condensation appears on the isotherm, accompanied by a characteristic H4-type hysteresis loop. The pronounced wide hysteresis loop, classified as type IV according to IUPAC guidelines, reaffirms the uniform pore size and mesoporous nature of the materials.



**Figure 14** –  $N_2$  adsorption-desorption isotherms of catalysts: a)  $AC_m$ ; b)  $5Pd/AC_m$ ; c)  $10Cu/AC_m$ ; and d)  $3Pd-7Cu/AC_m$ .

### 3.7. Catalytic hydrodechlorination of chlorobenzene and 1,2-dichlorobenzene

The  $5Pd/AC_m$ ,  $10Cu/AC_m$  and  $3Pd-7Cu/AC_m$  catalysts were tested using the model hydrodechlorination reactions of chlorobenzene and 1,2-dichloroben-

zene, separately, in 10 ml ethanol, at a temperature of  $50^\circ C$  and  $P_{H_2}$  of 10 atm, over a period of 5 hrs. The data on the catalytic activity for the neutralization of the organohalogen substances are given in Table 5 and Figures 15-17.

**Table 5** – Hydrodechlorination of chlorobenzene and 1,2-dichlorobenzene, using the  $5Pd/AC_m$ ,  $10Cu/AC_m$  and  $3Pd-7Cu/AC_m$  catalysts.

Sample	Chlorobenzene		1,2-dichlorobenzene	
	Conversionproduct	Conversion, %	Conversionproduct	Conversion, %
$5Pd/AC_m$	Benzene	82,90	Benzene	86,74
$10Cu/AC_m$	Benzene	70,20	Benzene	80,30
$3Pd-7Cu/AC_m$	Benzene	93,94	Benzene	89,79

To further contextualize these results, Table 6 compares the catalytic performance of the  $3Pd-7Cu/AC_m$  catalyst with representative Pd-Cu based systems previously reported in the literature. This

comparative overview clearly demonstrates that the Cu-rich  $3Pd-7Cu/AC_m$  composition provides competitive or even superior hydrodechlorination activity, while maintaining significantly lower Pd loading.

**Table 6** – Comparison of hydrodechlorination performance of Pd–Cu catalysts with previously reported systems.

Catalyst System	Metal Loading (wt%)	Support Type	Chlorobenzene Conversion (%)	1,2-Dichlorobenzene Conversion (%)	Main Products	Findings
$3Pd-7Cu/AC_m$	3 Pd, 7 Cu	HCl-modified activated carbon	93.94	89.79	Benzene	Highest conversions with reduced Pd loading due to Cu-rich nanoalloy and modified support

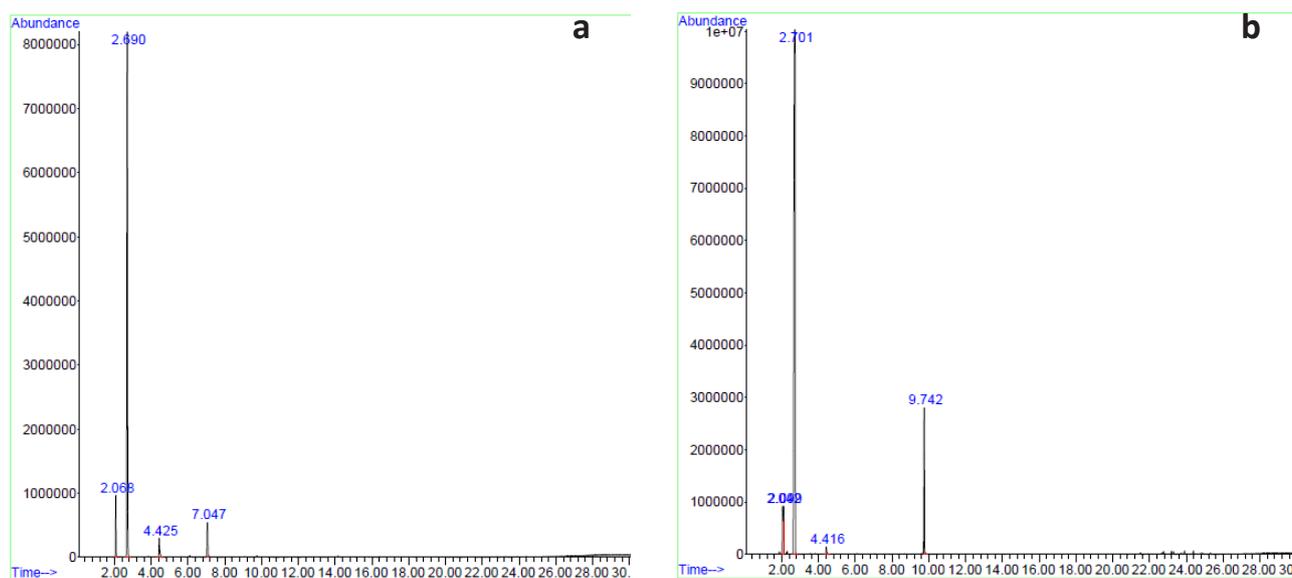
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Catalyst System	Metal Loading (wt%)	Support Type	Chlorobenzene Conversion (%)	1,2-Dichlorobenzene Conversion (%)	Main Products	Findings
5Pd/AC <sub>m</sub>	5 Pd	HCl-modified activated carbon	82.90	86.74	Benzene	Monometallic Pd catalyst, moderate conversions
10Cu/AC <sub>m</sub>	10 Cu	HCl-modified activated carbon	70.20	80.30	Benzene	Cu catalyst shows lower conversion compared to Pd or bimetallic
Pd-Cu/Coal-Based AC	~10 Pd	Coal-based activated carbon	80–100	-	-	Previously reported high activity with higher Pd content
Pd-Fe/GAC	Pd ~2-3 nm layer on Fe NPs	Granular activated carbon	~90 for PCB	-	Biphenyl	Bimetallic Pd-Fe catalysis, complex synthesis
Pd/C-Et3N	10 Pd	Modified carbon (triethylamine)	100 for PCB	-	Biphenyl	Complete decomposition of PCBs; high Pd loading
Cu/PVP-AC	~5 Cu	AC stabilized with PVP	94.46	-	Benzene	Copper-only catalyst with stabilizer shows high conversion
Pd/C (various)	10 Pd	Various activated carbons	80–100	-	Various	Widely studied Pd catalysts for hydrodechlorination with high activity

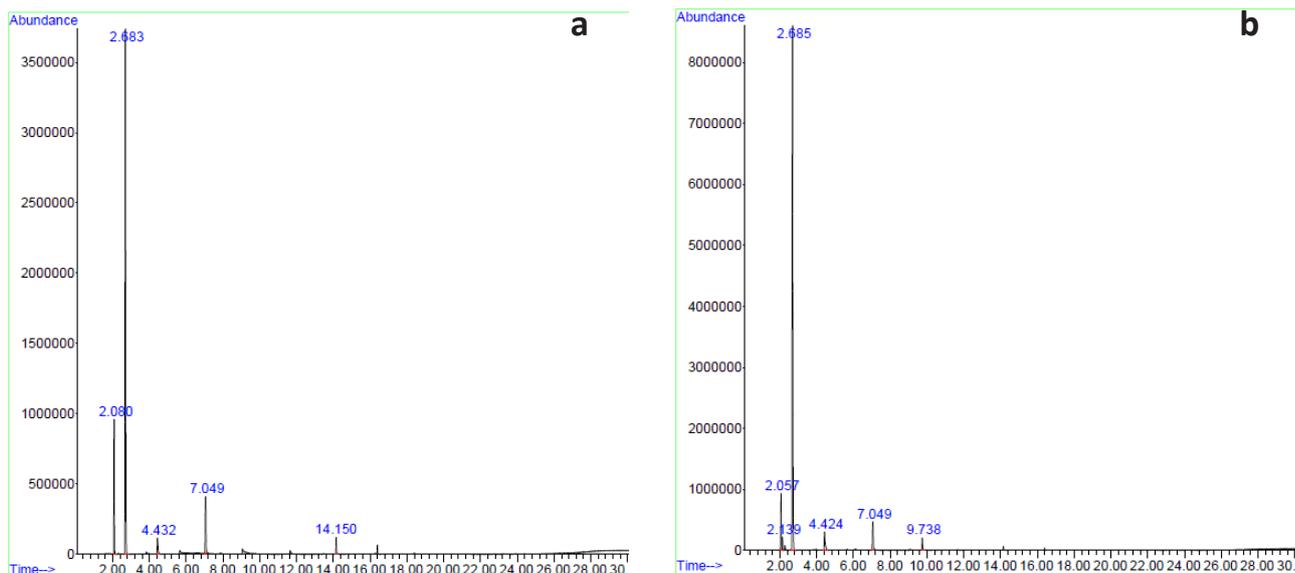
### 3.8. Gas chromatography-mass spectrometry analysis

The conversion of chlorobenzene through hydrodechlorination using 5Pd/AC<sub>m</sub>, 10Cu/AC<sub>m</sub> and 3Pd-7Cu/AC<sub>m</sub> is 82.9%, 70.20 and 93.94%, respectively (Table 5, Figures 14a-16a). Hydrodechlorination of 1,2-dichlorobenzene using 5Pd/AC<sub>m</sub>, 10Cu/AC<sub>m</sub> and 3Pd-7Cu/AC<sub>m</sub> is 86.74%, 80.30% and 89.79%, respectively (Table

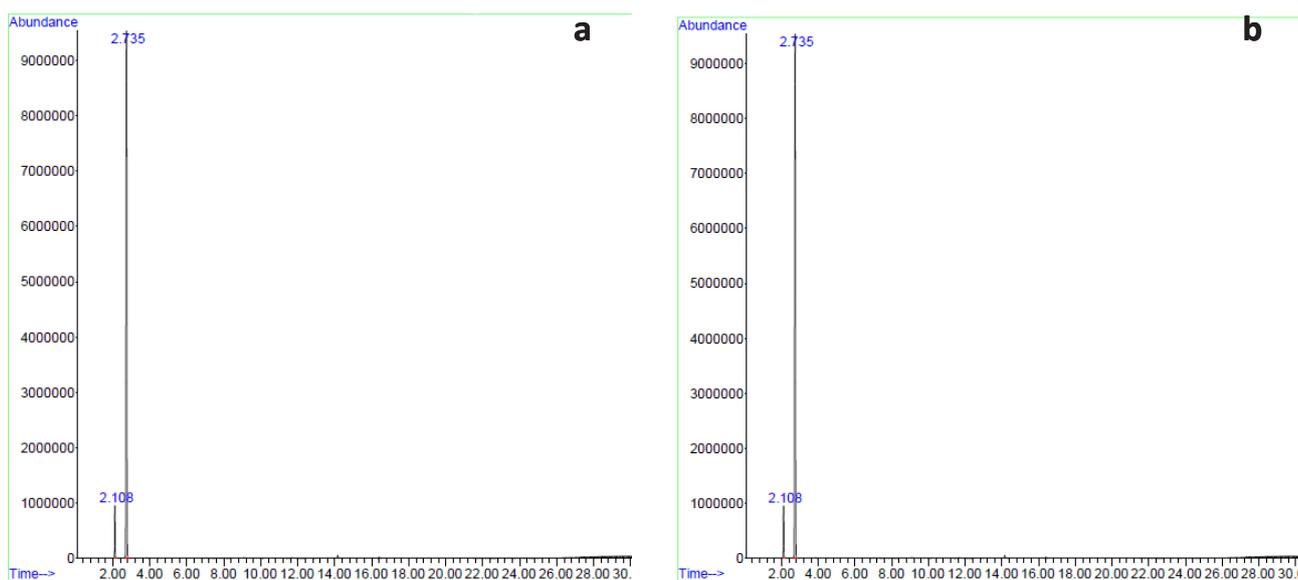
5, Figures 14b-16b). In both cases, the conversion product was benzene. The bimetallic catalyst, 3Pd-7Cu/AC<sub>m</sub>, showed the greatest activity in the hydrodechlorination of both organohalogens, with a conversion rate of 93.94% and 89.79%, respectively. The order of increasing catalytic activity in the hydrodechlorination of organohalogen substances is 10Cu/AC<sub>m</sub> < 3Pd/AC<sub>m</sub> < 3Pd-7Cu/AC<sub>m</sub>.



**Figure 15** – Chromatogram of the hydrodechlorination products of:  
a) chlorobenzene; b) 1,2-dichlorobenzene, using 5Pd/AC<sub>m</sub> (10 mg) at a temperature of 50°C.



**Figure 16** – Chromatogram of the hydrodechlorination products of:  
a) chlorobenzene; b) 1,2-dichlorobenzene, using 10Cu/AC<sub>m</sub> catalyst (10.0 mg) at a temperature of 50°C.



**Figure 17** – Chromatogram of the hydrodechlorination products of:  
a) chlorobenzene; b) 1,2-dichlorobenzene, using 3Pd-7Cu/AC<sub>m</sub> catalyst (10 mg) at a temperature of 50°C.

#### 4. Conclusions

Three dehydrochlorination catalysts were synthesized on modified activated carbon (AC<sub>m</sub>), using Pd and Cu nanoparticles (NPs), both alone and as a bi-metal. Based on prior research, the following combinations were selected, using estimations of both cost and effectiveness: 5% Pd (5Pd/AC<sub>m</sub>), 10% Cu (10Cu/AC<sub>m</sub>), and 3% Pd with 7% Cu (3Pd-7Cu/AC<sub>m</sub>). As per FTIR, TGA and EDX analyses, the NPs are able

to connect with the AC<sub>m</sub> carrier at the carboxyl and carbonyl functional groups. The pore sizes of the resulting catalysts have a range of 19.070 to 19,168 nm, which is 100 times smaller than that of the AC<sub>m</sub> carrier alone. Further, the surface areas of the catalysts were found to be in the range of 371,748 to 672,132 m<sup>2</sup>/g, which is 1.6-2.9 times higher than that of AC<sub>m</sub>. These results on the pore structures of the catalysts demonstrate good development, allowing for an increased number of sites to adsorb persistent organic

pollutants (POPs). Whereas all the catalysts showed effectiveness in dehydrochlorinating chlorobenzene and 1,2-dichlorobenzene, separately, into benzene, the bimetallic catalyst, 3Pd-7Cu/AC<sub>m</sub>, demonstrates the best results, with conversion rates of 93.94% and 89.79%, respectively.

The usage of inexpensive metallic NPs, like Cu, as catalysts on an AC<sub>m</sub> carrier allows for a reduction in the amount of more expensive materials, such as Pd. The need for efficient and cost-effective catalysts to decrease the number of POPs is of great importance to all nations, especially in Kazakhstan, which faces serious problems created by both the industrial and agricultural sectors regarding POPs. This is in accordance with the WHO Convention on Persistent Organic Pollutants, which targets the reduction of both existing and future halogenated organic compounds. Furthermore, as Kazakhstan is already known for its petroleum production, the use of new types of nanocatalysts that regenerate the hydrocarbon component of halogenated molecules can create a synergistic relationship among the petroleum, agricultural, chemical and research sectors of the country. Finally, the current research team hopes to particularly find opportunities for this technology in the cleanup of hazardous areas that have been negatively affected by both past and recent application of halogenated organic compounds.

A deeper mechanistic explanation of the catalytic pathway can be formulated by considering the specific roles of Pd, Cu, and the HCl-modified carbon surface. According to our FTIR results, both Pd and Cu nanoparticles are bound to carboxyl and carbonyl functionalities of the modified activated carbon. This anchoring not only stabilizes the nanoparticles, but also electronically couples them to the support, creating an interface that facilitates H<sub>2</sub> activation and stabilizes surface hydride species. XRD analysis confirms the formation of Pd-Cu alloy phases, which is a direct structural indicator of electronic interaction between the two metals at the atomic level. In Pd-Cu nanoalloys, the presence of Cu modifies the electronic density of Pd: electron donation from Cu to Pd shifts the d-band center of Pd to lower energy, which is known

to weaken the Pd-Cl interaction. As a result, the C-Cl bond cleavage pathway becomes energetically more favorable, and surface poisoning by strongly adsorbed chlorides is reduced. Pd remains the primary site for H<sub>2</sub> dissociation, while Cu facilitates electron transfer steps and stabilizes reactive intermediates, which together increases the overall turnover for hydrodechlorination. The modified carbon support additionally contributes by providing high surface area mesoporosity and a high density of oxygen-containing groups, which improves nanoparticle dispersion and suppresses sintering or growth during reaction.

Thus, the high activity of the 3Pd-7Cu/AC<sub>m</sub> catalyst can be explained mechanistically by the combination of (I) alloy-driven electronic tuning of Pd by Cu, (II) more efficient H<sub>2</sub> activation-transfer sequence at the bimetallic interface, and (III) stronger metal-support interactions induced by HCl modification of the carbon. These three effects act synergistically, and this explains why a Cu-rich formulation with reduced Pd loading can achieve higher conversion than monometallic Pd or Cu catalysts.

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