Research on the fabrication and evaluation of certain properties of the multi-metallic catalyst supported on silica TeKL-25

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ABSTRACT

This paper presents the research results on the synthesis of the multi-metallic catalyst material TeKL-25 based on a silica support. The characteristics of the synthesized material were analyzed using advanced techniques such as Field Emission Scanning Electron Microscopy (FE-SEM), Brunauer-Emmett-Teller (BET) surface area analysis, X-ray Diffraction (XRD), and Energy Dispersive X-ray Spectroscopy (EDX). The synthesized TeKL-25 material exhibits a tightly bonded structure, a rough surface, relatively uniform particle distribution, a BET surface area of 292.716 m^2/g*, and a mesoporous structure.*

Keywords: Multi-metallic catalyst; Silica; TeKL-25.

1. INTRODUCTION

Multimetallic catalysts are an advanced class of catalysts synthesized by combining two or more metals to enhance catalytic activity, selectivity, and durability. The structure of multimetallic catalysts generates significant synergistic effects, where each metal plays a distinct role in forming active catalytic sites, reducing activation energy, and optimizing complex chemical processes. The diversity in composition and structure of multimetallic systems allows for flexible tuning of catalytic properties, meeting technical requirements across various fields such as petrochemistry, organic synthesis, and energy technology [1, 2].

The CuMnFe catalyst supported on TEOS (tetraethyl orthosilicate) is an advanced catalytic system developed to optimize oxidation reactions and environmental treatment processes. In this catalyst, copper (Cu), manganese (Mn), and iron (Fe) are dispersed on a silica matrix derived from the TEOS precursor, forming a structure with high specific surface area and stability, which enhances active catalytic sites. The TEOS support not only provides structural reinforcement but also improves the thermal stability of the metals, thereby maintaining catalytic activity during operation [2].

Thanks to the synergistic interaction between Cu, Mn, and Fe, this catalyst can generate highly reactive oxygen species (e.g., hydroxyl radicals *OH and superoxide O_2^- with strong oxidative potential), effectively decomposing organic pollutants and recalcitrant compounds in aqueous and gaseous environments [2]. This combination offers significant advantages in terms of selectivity, durability, and conversion efficiency, making CuMnFe/TEOS an attractive choice for various applications, including wastewater treatment, air purification, chemical synthesis, and clean energy technology [3].

In multimetallic catalytic systems, silica is widely used as a support material due to its high specific surface area, excellent thermal stability, and effective dispersion of metal nanoparticles. Multimetallic catalysts on silica supports enable uniform metal distribution, thereby increasing surface contact and catalytic activity while minimizing catalyst poisoning and sintering. With high application potential and low cost, CuMnFe catalysts supported on TEOS have become a promising research focus in industrial and environmental fields. They have demonstrated high efficiency in critical applications such as exhaust gas treatment, wastewater remediation, cracking and hydrocracking in the petroleum refining industry, and environmentally friendly production processes [4].

Due to their outstanding properties, these catalytic systems are being further investigated not only to enhance industrial process efficiency but also to meet sustainability requirements in science and technology. To contribute to the research and development of multimetallic catalysts on silica supports for the degradation of organic compounds in wastewater, this study focuses on optimizing the structure and surface properties of the TeKL-25 material to enhance catalytic performance.

2. MATERIALS AND METHODS

2.1. Chemicals and equipment

a. The chemicals used in the experiment include:

- Tetraethyl orthosilicate, $C_8H_{20}O_4Si$, 98%, Macklin, China;
- Triethanolamine, $C_6H_{15}NO_{3,}$ ≤ 100%, Xilong Scientific, China;
- Triethylamine hydrochloride, $C_6H_{16}ClN$, 98%, Macklin, China;
- Iron (II) sulfate heptahydrate, $FeSO_4.7H_2O$, 99%, Guangdong, China;
- Iron (III) chloride hexahydrate, $FeCl_3.6H_2O$, ≥ 99%, Xilong Scientific, China;
- Manganese (II) acetate tetrahydrate, $Mn(CH_3COO)_2.4H_2O$, ≥ 99%, Xilong Scientific, China;
- Copper (II) acetate monohydrate, $Cu(CH_3COO)_2.H_2O$, ≥ 99%, Xilong Scientific, China;
- Sodium hydroxide, NaOH, 97%, Fisher Scientific, USA;
- Ultra-pure water, Milli-Q, Merck Millipore, France.

b. The equipment used in the experiment includes:

- Electric Therm furnace (t_{max}=1500 ^oC), Viet Nam;
- Value Vacuum Machine, China;
- Autoclaver 45 mL, USA;
- DLAB Magnetic Stirrer with Heating, USA;
- GT Sonic Ultrasonic Cleaner, China;
- Some other experimental equipment and instruments are available in the laboratory of Institute of New Technology.

2.2. Methods

2.2.1. Study on the fabrication process of multimetallic catalysts supported by silica

The fabrication process was carried out and repeated three times to ensure accuracy and reproducibility. After synthesis, the obtained samples were thoroughly mixed to achieve homogeneity before conducting subsequent structural and surface characterization studies.

A total of 0.01 mol of $FeCl_2·4H_2O$ and 0.02 mol of $FeCl_3·6H_2O$ were dissolved in 100 mL of deionized water in a 500 mL three-necked round-bottom flask. Subsequently, 100 mL of 0.8 M NaOH solution was added dropwise into the reaction mixture under continuous stirring at 500 rpm for 3 hours at room temperature, while purging with nitrogen gas to maintain an inert atmosphere. Upon completion of the reaction, the resulting solid was collected by centrifugation at 1500–2000 rpm, followed by multiple washings with ultrapure water until the complete removal of chloride ions was confirmed by the absence of a precipitate when tested with 0.01 N $AgNO_3$ solution. The washed product was dried at room temperature in a silica gel desiccator until constant weight, then stored in a sealed amber glass vial protected from light. Prior to incorporation into the TEOS-based matrix, the black Fe_3O_4 nanoparticles were ground using a planetary ball mill to achieve a particle size of less than 100 nm.

Magnetite (Fe_3O_4) nanoparticles were synthesized using the co-precipitation method from an iron salt solution in a strongly alkaline medium [5]. After synthesis, 25 mg of nanoparticles were dispersed in 25 g of tetraethyl orthosilicate (TEOS) with a Fe/Si molar ratio of 0.01. The dispersion process was performed using ultrasonic treatment for 30 minutes at room temperature to ensure system homogeneity.

Next, manganese(II) acetate tetrahydrate ($Mn(CH_3COO)_2{\cdot}4H_2O$) and copper(II) acetate monohydrate ($Cu(CH_3COO)_2{\cdot}H_2O$) were introduced into the system at Mn/Si and Cu/Si molar ratios of 0.01. The mixture was stirred for 10 minutes to ensure the uniform distribution of metal ions within the sol-gel solution.

Subsequently, triethanolamine (TEA, 8.86 g) was used as a structure-directing agent to facilitate silicate condensation. TEA was dissolved in 16.4 g of distilled water and added to the reaction system, followed by continuous stirring for 30 minutes at room temperature. To adjust the pH of the mixture, 8.66 g of tetraethylammonium hydroxide (TEAOH, 20%, Acros) was weighed and added to the system to regulate the hydrolysis and condensation of the silicate precursor, the final pH value of the solution ranged from 9 to 10. The resulting gel was aged overnight at room temperature before being dried at 50 °C for 24 hours to remove excess solvents.

The dried material was then subjected to hydrothermal treatment in an autoclave (50 mL capacity) at 150 °C for 48 hours, using ethanol as the reaction medium to control system crystallization. The obtained solid product was separated by decantation and filtration, followed by multiple washing steps with ethanol and water to remove organic impurities. The material was subsequently dried at room temperature overnight until a constant weight was achieved and stored in dark glass bottles with airtight lids to minimize moisture absorption and prevent structural degradation.

The final stage involved solvent extraction in ethanol at 80 °C overnight to remove residual structure-directing agents, followed by calcination at 450 °C for 6 hours in an air atmosphere. The heating rate was controlled at 1 °C/min to prevent uneven pore shrinkage. The synthesized material was designated as TeKL-25.

2.2.2. Evaluation of some properties of TeKL-25 material after fabrication

The surface morphology and particle size of the material were analyzed using a field emission scanning electron microscope (FE-SEM, Hitachi S-4800, Japan). The chemical composition and structural characteristics of the material were determined by energy-dispersive X-ray spectroscopy (EDX) using an Oxford Instrument system (USA). These analyses were conducted at the Institute of Materials Science, Vietnam Academy of Science and Technology.

The porosity of the material, including surface area and pore size distribution, was measured using the BET method on a TriStar II 3020 system (Micromeritics, USA). The phase composition and functional groups were identified using X-ray diffraction (XRD) on a Panalytical instrument (Netherlands). These measurements were performed at the Institute of Chemistry and the Institute for Tropical Technology, both under the Vietnam Academy of Science and Technology.

3. RESULTS AND DISCUSSION

3.1. Research results on the fabrication of TeKL-25 material

The synthesized material exists in the form of a dry, powdery substance with discrete, non-adhesive particles, exhibiting a brown color mixed with light gray. The particle sizes are non-uniform, consisting of both small and large particles, with some dispersed around the material. The surface appears rough, with sharp edges and possibly small pores, indicating porosity and a high surface area, making it suitable for applications such as catalysis or adsorption. The synthesis yield achieved 68-72% compared to the theoretical calculation.

3.2. Surface morphology and particle size of TeKL-25 material

FE-SEM images of the TeKL-25 material at different magnifications are presented in figure 2.

At the highest magnification (figure 2a, 200,000×), nanoscale particles with irregular shapes and a tendency to aggregate into clusters can be observed. The porous structure of the material is also clearly visible, indicating the presence of mesopores. When the magnification is reduced to 100,000× (figure 2b), particle aggregation becomes more pronounced, while the material's surface still exhibits high porosity.

Figure 1. *TeKL-25 material.*

At an intermediate magnification (figure 2c, 50,000×), the material displays a network-like porous structure, where particle clusters are interconnected, forming mesoporous regions. The particle size distribution suggests that the material can provide a large surface area, which is beneficial for adsorption and catalytic reactions. When the magnification is further decreased to 5,000× (figure 2d), the formation of micron-sized particle clusters becomes more apparent, with a tightly interconnected structure and a rough surface. This indicates that the material has good mechanical stability while maintaining porosity to facilitate reactant diffusion.

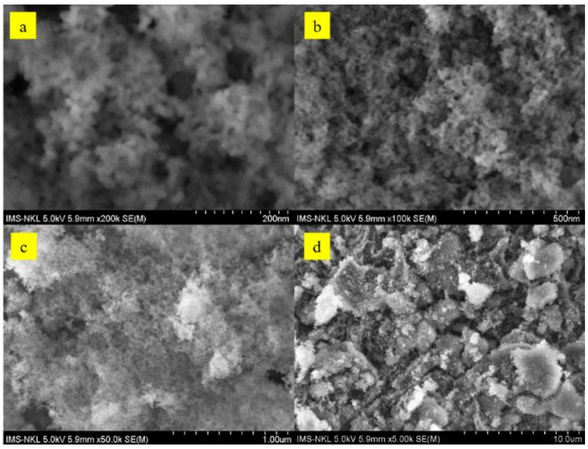


Figure 2. *FE-SEM images of the TeKL-25 material.*

Comparing the FE-SEM images, it can be observed that at high magnifications (figures 2a, 2b), the nanoparticles are uniformly distributed on the silica support, whereas at lower magnifications (figures 2c, 2d), the aggregated structure of the material becomes more evident. This suggests that the synthesized material exhibits a typical mesoporous structure, combining nanoscale particle size with a mesoporous network, making it suitable for heterogeneous catalysis applications. These findings are consistent with the published research results of Do Thi Phuong Hoang et al. [6].

3.3. Structural characterization of TeKL-25 material

3.3.1. X-ray diffraction (XRD) analysis results

The X-ray diffraction (XRD) analysis results of the Fe-Mn-Cu/Silica (TeKL-25) material after synthesis are presented in figure 3.

The obtained results show a broad diffraction peak at approximately 2θ~22°-25°, characteristic of the amorphous silica (SiO_2) matrix. However, no sharp diffraction peaks corresponding to the metal oxide phases of Fe, Mn, and Cu were observed. This is likely due to the extremely small size of the metal or metal oxide components, their existence in an amorphous state, or their dispersion at the nanoscale within the silica network.

These findings are consistent with previously published studies. Specifically, in the research by Khalameida et al., transition metal oxides were dispersed on a mesoporous silica support using the impregnation method. XRD analysis results showed that the deposited oxides exhibited an amorphous or poorly crystalline structure, with no sharp diffraction peaks corresponding to metal oxide phases. This suggests that the metal oxides may exist in an amorphous state or as nanoscale dispersions within the silica network, similar to the results reported in previous studies [7].

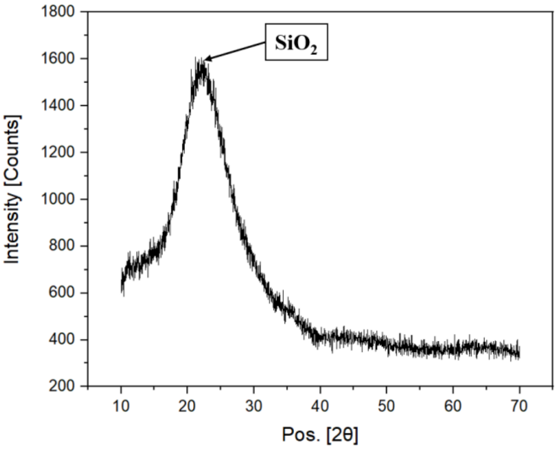


Figure 3. *XRD diagram of TeKL-25 material.*

Additionally, research by Coombes et al. also indicated that during the reduction of iron oxides promoted by silica, the iron oxide phases could exist in an amorphous form or at an extremely small size, leading to the absence of distinct diffraction peaks in the XRD spectra [8].

These studies support the explanation that the “absence” of characteristic diffraction peaks for metal oxide phases in the TeKL-25 sample may be due to the metal oxides existing in an amorphous form or being dispersed at the nanoscale within the silica network, resulting in weak diffraction signals or signals below the detection limit of the XRD method.

Furthermore, the lack of characteristic peaks for metal oxide phases may be attributed to the low metal concentration in the material, leading to weak diffraction signals that fall below the detection threshold of XRD. Additionally, interactions between metal ions and the silica matrix may reduce the crystallinity of the metal oxide phases, making them difficult to detect using conventional XRD techniques. This result aligns with previous studies on metal-based catalysts supported on silica, where metal ions can exist as amorphous dispersions or clusters too small to generate sufficiently strong diffraction signals.

3.3.2. Energy-dispersive X-ray spectroscopy (EDX) results

Based on the EDX elemental distribution images of the multi-metal catalyst TeKL-25 (figure 4), the analysis is as follows:

The EDX images illustrate the elemental distribution of the Fe-Cu-Mn multi-metal catalyst on the silica support. The uniform distribution of Si and O indicates that the silica matrix plays a crucial role in the material’s structure, ensuring a sufficiently large surface area and facilitating the dispersion of metal phases.

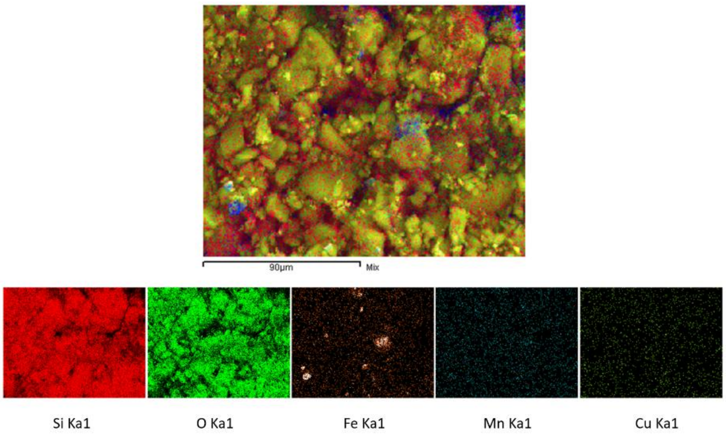


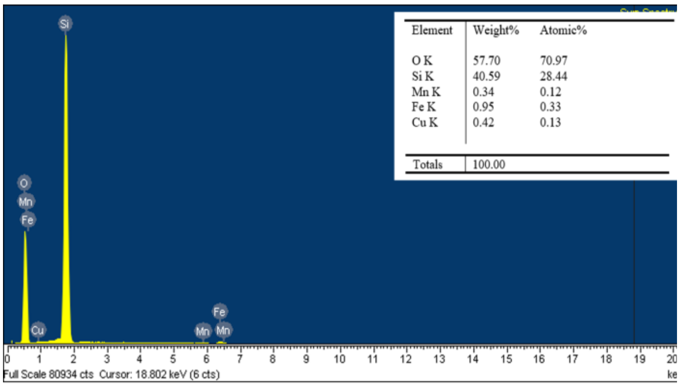
Figure 4. *EDX mapping of TeKL-25 material.*

The presence of Fe, Cu, and Mn is observed through their respective elemental maps. Fe tends to form localized clusters, suggesting possible aggregation or the formation of a separate iron oxide phase. Mn appears to be more evenly dispersed but at a lower density, indicating its incorporation into the silica matrix, possibly as MnO_x species. Cu exhibits a scattered distribution with small particle sizes, likely existing as CuO or metallic Cu^0. The variations in metal phase dispersion suggest differing interactions between the metals and the silica matrix, which may significantly influence the catalytic activity.

These results are consistent with previous studies on metal catalysts supported on silica. Specifically, the study conducted by Hui-Ya Shih et al. in 2023 on the Cu-Fe/silicate system demonstrated that Cu and Fe metals were evenly distributed within the silica framework, supporting the conclusion of strong metal-support interactions [9]. Additionally, the study by Li et al. published in 2020 on the Cu-Mn system on silica also reported a uniform distribution of metals, aligning with the observations of this research [10]. Notably, the study by Wang et al. in 2022 on the Cu-Fe-Mn trimetallic catalyst system on mesoporous silica showed that these metals were homogeneously dispersed on the silica surface, similar to the findings of this study [11].

The EDX analysis confirms the presence of the key elements in the TeKL-25 catalyst material, including Si, O, Fe, Mn, and Cu. The highest-intensity peak corresponds to Si, reflecting the dominance of the silica matrix in the material composition, which aligns with its role as a support. Oxygen also exhibits a strong signal, indicating the presence of metal oxides such as SiO_2, Fe_2O_3, MnO_x, and CuO.

The peaks corresponding to Fe, Mn, and Cu have significantly lower intensities than Si, suggesting that these metals exist as dispersed phases on the silica support. The presence of Fe is identified by peaks around 6.4–7.1 keV, while Mn is observed through characteristic peaks in the 5.9–6.5 keV range. Cu exhibits a distinct peak at approximately 0.9 keV, corresponding to Cu Kα. The relatively low intensity of the Fe, Mn, and Cu peaks indicates that these metals may be present at low loading levels or highly dispersed within the silica matrix, which could influence the catalytic activity of the material.



Element	Weight%	Atomic%
O K	57.70	70.97
Si K	40.59	28.44
Mn K	0.34	0.12
Fe K	0.95	0.33
Cu K	0.42	0.13
Totals	100.00	

Figure 5. *EDX spectrum of TeKL-25 material.*

These results are consistent with the study by Rajabzadeh and Sardarian, published in 2024, in which the EDX spectrum of the Fe_3O_4@SiO_2-Pr-DEA-[NTA-Cu(II)]$_2$ material exhibited characteristic peaks of Fe, Si, O, and Cu, with the highest peak intensity for Si, reflecting the dominance of the silica matrix in the material [12].

Additionally, the study by Abdollahi-Alibeik and Ramazani, published in 2022, on the Fe_3O_4@Fe–Cu/MCM-41 material also demonstrated EDX peaks corresponding to Fe, Cu, Si, and O, with the Si peak showing a higher intensity compared to the other metals, indicating the dispersion of metals on the silica support [13].

These findings further support the conclusion that, in metal catalyst systems supported on silica, the metals often exist in a dispersed form with low concentrations, while the silica matrix remains the predominant component. This distribution can significantly influence the catalytic activity of the material.

The FE-SEM and EDX images reveal that the metal particles are uniformly distributed across the catalyst support surface, exhibiting nanoscale size and well-developed porous morphology. This is expected to facilitate the accessibility of reactants to the active sites, while also increasing the effective contact area between the catalyst and the reactants, thereby enhancing the overall catalytic performance.

3.4. HR-TEM micrograph of the material

The HR-TEM image reveals that the material predominantly exhibits an amorphous structure, which is characteristic of silica derived from the hydrolysis and condensation of TEOS. No large metallic clusters or well-defined crystalline domains are observed across the surveyed regions, suggesting that the amount of metal incorporated into the material is extremely low. In fact, with a molar ratio of Fe/Si around 0.01, the iron content is minimal, likely leading to either molecular-level dispersion or ultrafine metallic/metal oxide clusters that are below the resolution limit of conventional HR-TEM. A few short and scattered lattice fringes are detected in isolated areas, which may indicate the presence of nanoclusters or small crystalline domains of metal or metal oxides. However, the very low density of these fringes and the absence of an extended crystalline lattice further support the hypothesis that iron species are highly dispersed, potentially existing in the form of single atoms.

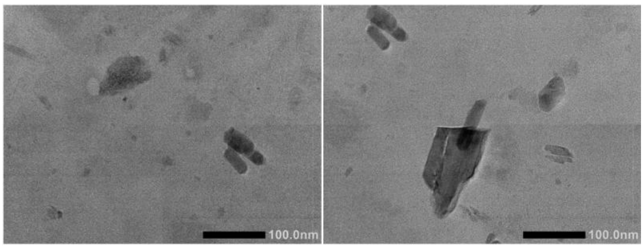


Figure 6. *The HR-TEM images of TeKL-25 material.*

Furthermore, the magnetic nature of the material presents additional challenges during sample preparation for HR-TEM analysis. The presence of magnetic components can cause the sample to be attracted unevenly to the TEM grid, resulting in non-uniform distribution or instability under the electron beam. This could significantly hinder the visualization of small or highly dispersed metal species. Nevertheless, despite these limitations, the HR-TEM observations remain a valuable piece of evidence, supporting the structural characteristics consistent with the design of highly dispersed, metal-loaded catalysts on a silica-based matrix.

3.5. The specific surface area of the material

The BET measurement results of the synthesized TeKL-25 material, presented in figure 7, illustrate the relationship between adsorption capacity and relative pressure. The specific surface area of TeKL-25 is 292.716 m^2/g, with a pore volume of 1.477 cm^3/g according to the adsorption method. Meanwhile, the micropore volume determined by the t-plot method is only 0.047 cm^3/g, indicating that the proportion of micropores in the total pore volume is negligible.

Observing figure 7, it can be noted that the graph closely resembles a type IV isotherm according to the IUPAC classification, which is characteristic of mesoporous materials with pore sizes ranging from 2 nm to 50 nm. This is a crucial indicator, as the presence of a hysteresis loop between the adsorption and desorption curves suggests capillary condensation, a common phenomenon in porous materials [14].

The pore size distribution results (inset in figure 7) indicate that the material exhibits a mesoporous structure, with the main distribution range between 4–8 nm, along with a minor proportion of larger pores (~15 nm). This relatively broad distribution has a dual impact on the transport characteristics within the material. The smaller mesopores contribute to a higher surface area, which is favorable for adsorption and reactions involving small to medium-sized molecules. Meanwhile, the presence of larger pores serves as auxiliary diffusion channels, facilitating deeper penetration and improved accessibility of reactants into the internal structure—particularly beneficial for catalytic or adsorption processes involving bulky molecules.

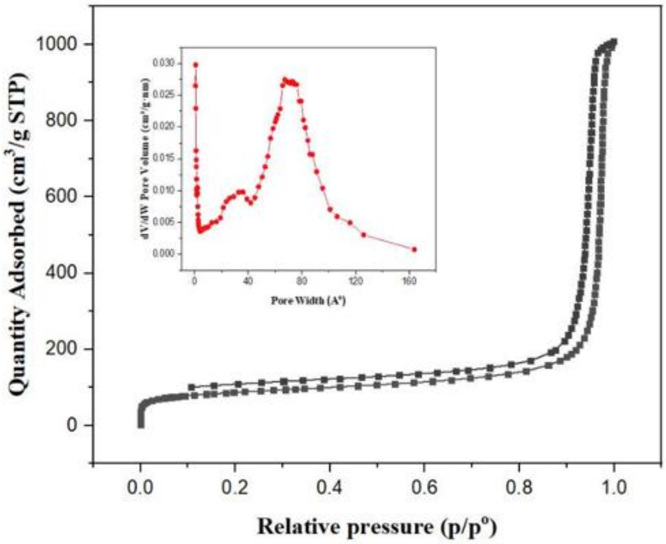


Figure 7. *Adsorption isotherm and pore size distribution of TeKL-25 material.*

This combination can be considered a potential advantage of the material, as it preserves a high surface area while ensuring efficient diffusivity- an essential factor for performance in heterogeneous catalysis, selective separation, or material storage systems.

At low relative pressure ($p/p° < 0.2$), the amount of adsorbed gas increases slowly, indicating monolayer adsorption on the material's surface. This stage reflects the interaction between the gas and the material's surface, typically associated with small pores or flat surfaces. As the pressure increases ($p/p° > 0.4$), the adsorption capacity rises significantly, suggesting multilayer adsorption and the onset of capillary condensation in larger pores. At high-pressure regions ($p/p° > 0.8$), a sharp increase in gas adsorption is observed, confirming the material's ability to retain gas within large pores and highlighting pronounced capillary condensation phenomena [15].

The observed hysteresis loop in the adsorption and desorption curves indicates that the adsorption and desorption mechanisms are not entirely identical, often due to differences in the pressure required for gas condensation and release from the pores, which is characteristic of mesoporous materials. This result aligns with previous studies, such as the research by Neimark et al. on capillary condensation in MCM-41 materials, where the disparity between adsorption and desorption curves formed a distinct hysteresis loop, reflecting the mesoporous structure of the material [16]. Additionally, the study by Jaroniec et al. demonstrated that the shape of the hysteresis loop can be used for a more detailed characterization of the mesoporous region of materials [17].

Thus, from the BET analysis, it can be concluded that the synthesized TeKL-25 material possesses a mesoporous structure with excellent gas adsorption capacity and well-defined capillary condensation characteristics. The BET evaluation confirms that the pore type of TeKL-25 is consistent with previously reported studies. This research successfully developed a multi-metal catalyst on a silica support, TeKL-25, with superior structural properties compared to traditional single-metal silica-supported catalysts or materials with lower surface areas [18]. Specifically, TeKL-25 exhibits a high specific surface area of 292.716 m^2/g, significantly higher than many previously reported mesoporous catalyst materials (typically < 200 m^2/g), which enhances the contact between reactive phases and improves catalytic efficiency. Furthermore, the optimized pore size and uniform distribution of metal nanoparticles improve reactant diffusion, minimizing pore blockage, a common issue in many conventional catalytic materials.

4. CONCLUSIONS

The research results indicate that the synthesized material possesses a tightly bonded structure, a rough surface, and a uniform distribution of nanoparticles, contributing to enhanced durability and stability in reaction environments. With these characteristics, TeKL-25 shows promising potential for applications in the treatment of hazardous organic compounds in wastewater, particularly nitramine compounds, due to its high adsorption capacity and catalytic activity. Advanced analytical techniques such as FE-SEM, BET, XRD, and EDX have provided a comprehensive understanding of the material's characteristics, enabling precise identification of structural phases and elemental distribution within the catalytic system. These findings not only open up practical application prospects for TeKL-25 but also make a significant contribution to the development of advanced mesoporous catalytic materials with high efficiency for environmental treatment processes and chemical synthesis.

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TÓM TẮT

Nghiên cứu chế tạo và đánh giá một số tính chất của vật liệu xúc tác đa kim loại trên nền silica TeKL-25

Bài báo giới thiệu kết quả nghiên cứu chế tạo vật liệu xúc tác đa kim loại trên nền silica TeKL-25. Đặc tính của vật liệu sau chế tạo được nghiên cứu thông qua một số phương pháp hiện đại như: Kính hiển vi điện tử quét (FE-SEM), diện tích bề mặt riêng (BET), quang phổ nhiễu xạ tia X (XRD), phổ tán xạ năng lượng tia X (EDX). Vật liệu TeKL-25 sau chế tạo có cấu trúc liên kết chặt chẽ, bề mặt nhám, các hạt phân bố tương đối đồng đều, giá trị BET đạt 292.716 m^2/g và kích thước mao quản trung bình.

Từ khóa: Xúc tác đa kim loại; Silica; TeKL-25.