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## MULTI-CORE SHELL NANOPARTICLES FOR EFFICIENT REMOVAL OF NITROPHENOLS FROM CONTAMINATED STREAMS

**Abstract.** This study aims to investigate the application of multi-core shell nanoparticles in heterogeneous Fenton processes for efficient removal of specific nitrophenols (2 and 4-nitrophenol) from contaminated wastewater. The synthesis of superparamagnetic magnetite (Fe<sub>3</sub>O<sub>4</sub>) was achieved through solution combustion synthesis (SCS) using a citric acid and iron nitrate solution. The mFe<sub>3</sub>O<sub>4</sub>@C catalyst was obtained via an adapted Stöber procedure. The material was characterized to identify specific chemical compositions related to the formation of the multi-core shell desired architecture. The carbon-coated catalyst exhibited superior performance compared to the bare core catalyst, leading to notable increases in the removal of 2-NP and 4-NP. Moreover, the decomposition of H<sub>2</sub>O<sub>2</sub> was significantly enhanced, indicating the system's potential in efficiently breaking down hazardous compounds. The findings of this study highlight the tremendous potential of the system as a highly effective and viable alternative for the efficient cleansing of contaminated streams polluted with 2-nitrophenol and 4nitrophenol.

**Keywords:** multi-core shell, nanoparticles, cwpo.



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**Introduction.** In recent years, industrialization and urbanization have led to a significant increase in pollution, posing a severe threat to the environment and human health [1-5]. Among the pollutants, nitrophenols, known for their toxic and potentially carcinogenic properties, are commonly found in wastewater from industries like pharmaceuticals, pesticides, and dyes [6-9]. However, traditional wastewater treatment methods often fail to effectively remove nitrophenols due to their complex nature and resistance to degradation. Therefore, there is a pressing need for innovative technologies to tackle this environmental challenge [10-14].

One promising approach is the use of multi-core shell nanoparticles in catalytic wet peroxide oxidation (CWPO) processes. This method involves generating highly reactive hydroxyl radicals ( $\cdot\text{OH}$ ) to degrade organic pollutants [15–19], with multi-core shell nanoparticles acting as catalysts. These nanoparticles have a well-defined structure consisting of a central core and an outer shell. The core stores catalytic species, while the shell provides stability and surface modification capabilities. Studies also reported an increase in the activity of the catalysts towards organic pollutants removal due to the activity of the carbon layer in the process. In this regard, there is a synergistic effect occurring by the carbon coating, that is both protecting the core against leaching and increasing the activity of the nanomaterial [20].

This study aims to investigate the application of multi-core shell nanoparticles in heterogeneous Fenton processes for efficient removal of specific nitrophenols (2 and 4-nitrophenol) from contaminated wastewater. The research will focus on synthesizing and characterizing the multi-core shell nanoparticles and discuss the removal results obtained. By advancing our understanding of multi-core shell nanoparticles' utilization in heterogeneous Fenton processes for nitrophenol removal, this research seeks to develop more efficient and sustainable wastewater treatment strategies. The outcomes of this study have the potential to pave the way for novel nanotechnology-based solutions to address the pressing challenges associated with nitrophenol contamination.

**Conditions and methods of research.** *Chemicals and apparatus.*

The following reactants were used in this study: iron (III) nitrate nonahydrate (99%, Sigma Aldrich, Germany), citric acid monohydrate (98.8%, WWR Chemicals, Belgium), ethanol absolute (99.8%, Fischer Chemical, South Africa), tetraethyl orthosilicate (TEOS, 98%, Fluka Chemika, Germany), 1,3-benzenediol (99%, Alfa Aesar, USA), methanal (37-38% wt.%, PanReac, Spain), ammonia solution 28 - 30% (Merck, Germany), sodium hydroxide (98.73%, Fisher Chemical, U.K.). Potassium bromide (>99 wt.%, Sigma Aldrich) was used to prepare pellets for Fourier transformed infrared analysis. Paracetamol (98 wt.%, Alfa Aesar), hydrogen peroxide (30% w/v, Fischer Chemical), titanium (IV) oxysulfate (99.99% w/v, Sigma Aldrich), sulfuric acid (98% v/v, Labkem), sodium sulfite (98 wt.%, Panreac), acetonitrile (99.9% v/v, Fisher Scientific), and orthophosphoric acid (85% v/v, Fisher Chemical) were used in CWPO runs and analytical techniques.

*Preparation of multi-core shell nanoparticles*

The synthesis of superparamagnetic magnetite ( $\text{Fe}_3\text{O}_4$ ) nanoparticles was achieved through solution combustion synthesis (SCS) using a citric acid and iron nitrate solution. The process involved heating the solution, followed by washing and drying the resulting product. The carbon-based multi-core shell architecture was developed in multiple stages: coating, carbonization, and etching. In the coating stage, a mixture of magnetite, distilled water, ethanol, resorcinol, and ammonia solution was stirred, and then formaldehyde and tetraethyl orthosilicate (TEOS) were added. The mixture was stirred and washed, resulting in the formation of the resin coated magnetite. For the carbonization step, the resin coated nanoparticles were annealed under a nitrogen atmosphere at various temperatures. In the final step, the nanoparticles were treated with a sodium hydroxide solution to remove the silica content, washed, and dried. The resulting material was denoted as  $\text{mFe}_3\text{O}_4@\text{C}$ . Overall, the synthesis involved a combination of solution combustion synthesis, coating, carbonization, and etching steps to produce the desired core-shell nanoparticles [21].

### *Characterization techniques*

The characterization techniques were performed according to reported in previous studies [22, 23]. The morphology of the nanoparticles, including their multi-core shell architecture and size were examined using Transmission Electron Microscopy (TEM) with a JEOL JEM 2100 operating at 200 kV. Fourier-transform infrared spectroscopy (FT-IR) was conducted on sample pellets prepared with KBr using a Perkin Elmer FT-IR spectrophotometer UATR Two to study the functionalities present in the samples. N<sub>2</sub> adsorption-desorption isotherms at 77 K were obtained using a Quantachrome NOVATOUGH LX<sup>4</sup> adsorption analyzer to analyze the textural properties of the nanoparticles. These characterization techniques provided valuable insights into the structural, chemical, and magnetic properties of the nanoparticles.

### *Liquid-phase oxidation experiments*

The CWPO (catalytic wet peroxide oxidation) experiments were conducted using a 250 mL round-bottom flask with 2-nitrophenol and p-nitrophenol with 50 mg L concentration. The temperature and pH were controlled, and the stoichiometric amount of hydrogen peroxide was added (696 mg L<sup>-1</sup>). The catalyst was added after the system was stirring for 5 min to achieve homogeneity, and this was marked as the beginning of the reaction. Samples were taken at specific time intervals for analysis. The concentration of pollutants and its oxidized intermediate products was measured using an HPLC system. H<sub>2</sub>O<sub>2</sub> concentration, total organic carbon (TOC) was determined using TOC-L analyzer. The presence of iron leaching was assessed using atomic absorption spectroscopy. Overall, these analytical techniques provided valuable information about the reaction progress and the formation of byproducts during the CWPO process.

### **Results and discussion.** *Characterization of multi-core shell nanoparticles.*

The core-shell structure of the developed nanoparticles was verified through representative transmission electron microscopy (TEM) images, shown in Figure 1. In the magnetic core, highly ordered crystalline domains were clearly observed, indicating the presence of a multi-core structure formed by smaller magnetite nanoparticles. The particle size was determined using ImageJ software ( $17.7 \pm 4.2$  nm) to be below the threshold for single-to-multi-domain transition, consistent with the observed superparamagnetic properties of the sample. The results confirm the amorphous carbon composition of the shell, as evidenced by the distribution of C throughout the shell region. Furthermore, the Fe and O spatial distribution is limited to the core volume, confirming the presence of an iron oxide core. The Si element is randomly distributed throughout the inorganic magnetic core, indicating interstitial occupation. To further support these findings, a quantitative EDX analysis was performed across the multi-core shell structure, as depicted in Figure 1 (b). The density of C was observed to be higher within the shell thickness compared to the core. On the other hand, the elemental density of Fe, O, and Si was localized exclusively within the core, with no presence detected in the outer carbon shell. These results, in conjunction with the structural and magnetic findings, solidify the confirmation of the multi-core shell structure of the nanoparticles.

Figure 2 displays the adsorption isotherms of N<sub>2</sub> at 77 K for the bare core and final sample, along with the results obtained for BET surface area, pore volume and pore diameter. According to the IUPAC classification of N<sub>2</sub> adsorption isotherms, these isotherms belong to type IV, characterized by a hysteresis loop commonly observed in mesoporous materials. The hysteresis loop observed in this study corresponds to type H4, indicating the presence of narrow slit-like pores. Each adsorption isotherm includes information on the textural properties of the materials.

Comparing the SBET results obtained for the material reveals a significant increase in surface area, from  $35 \text{ m}^2 \text{ g}^{-1}$  in the bare core to  $265 \text{ m}^2 \text{ g}^{-1}$ , which is ascribed to the removal of silica during the etching procedure. The removal of silica is fundamental to achieve the multi-core shell architecture since the removal of the intrastitial silica between the cores will promote the increase in the catalyst active sites. Other studies have also reported a similar behavior for synthesis of hybrid catalysts following the adapted Stöber procedure.

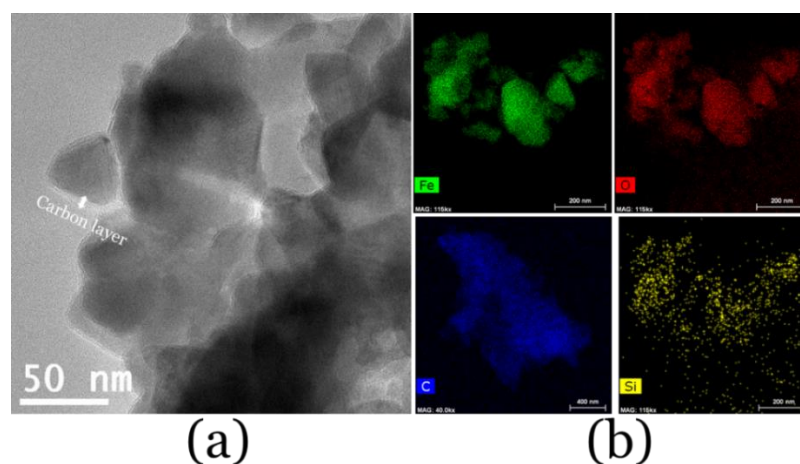


Figure 1. TEM image (a) and elemental mapping (b) of  $\text{mFe}_2\text{O}_3@\text{C}$  sample.

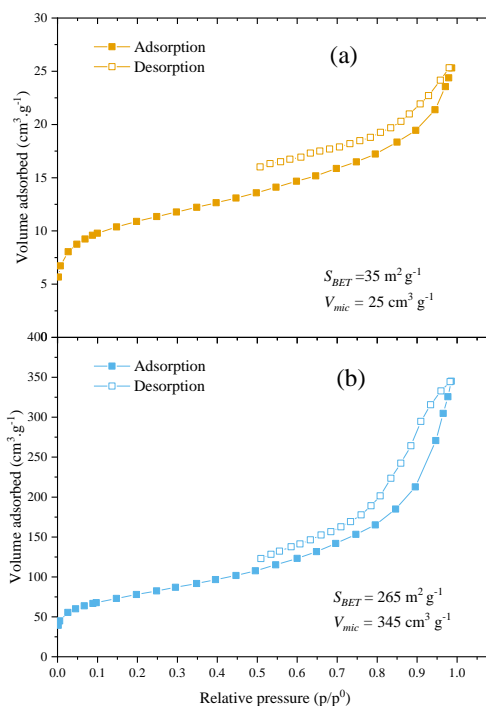


Figure 2.  $\text{N}_2$  isotherms, BET surface area and total pore volume for bare core (a) and  $\text{mFe}_2\text{O}_3@\text{C}$  (b).

To gain insights into the chemical bonds within the material's structure and their correlation with the synthesis treatments, Fourier-transform infrared spectroscopy (FT-IR) analysis was conducted. This analysis aimed to investigate the effects of functionalization on the surface properties of the material. Figure 3 illustrates a comparison of the FT-IR spectra obtained for bare core and final nanoparticles. The presence of magnetite was discerned from the absorption bands observed at  $564\text{ cm}^{-1}$ . Additionally, specific bands at  $1632$  and  $3436\text{ cm}^{-1}$  were identified, corresponding to the bending and stretching vibrations, respectively, of the surface hydroxyl (-OH) groups present in the carbon-based shell. These observations provide valuable information about the nature of the chemical bonds and the impact of functionalization on the material's surface.

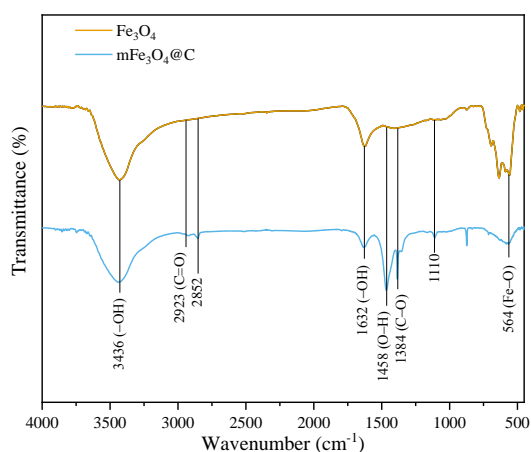


Figure 3. FT-IR results for bare core and final nanoparticle.

#### *Nitrophenols removal in simulated matrix*

The results obtained for  $\text{H}_2\text{O}_2$  decomposition and pollutant removal are depicted in Figure 4. The results of the study demonstrated that pollutant removal by adsorption is not significant since after an 8-hour duration,  $\text{Fe}_3\text{O}_4$  as an adsorbent achieved a modest removal of 3.1% for 2-NP and 5.6% for 4-NP. However, when  $\text{mFe}_3\text{O}_4@\text{C}$  was used as the adsorbent, the removal increased to 6.4% for 2-NP and 9.1% for 4-NP. Interestingly, the oxidation results depicted in Figure 1 provided valuable insights into the performance of the catalysts. The carbon-coated catalyst ( $\text{mFe}_3\text{O}_4@\text{C}$ ) exhibited superior performance compared to the bare core catalyst. After 8 hours, the removal of 2-NP increased by an impressive 17.7%, while 4-NP removal showed a notable enhancement of 10.1%. Moreover, the decomposition of  $\text{H}_2\text{O}_2$  was significantly improved, with a remarkable increase of 35.8% compared to the bare core catalyst. This enhanced activity of the carbon-coated catalyst can be attributed to the presence of the carbon surface derived from carbon coating process. The findings strongly suggest that oxidation reactions play a pivotal role in the removal of pollutants from the system. Furthermore, the increased adsorption and pollutant removal observed with the carbon-coated catalyst highlight the need for further investigation to determine if the degradation process was also influenced by changes in the catalyst's affinity with the pollutants, potentially due to alterations in lipophilicity caused by the carbon coating.

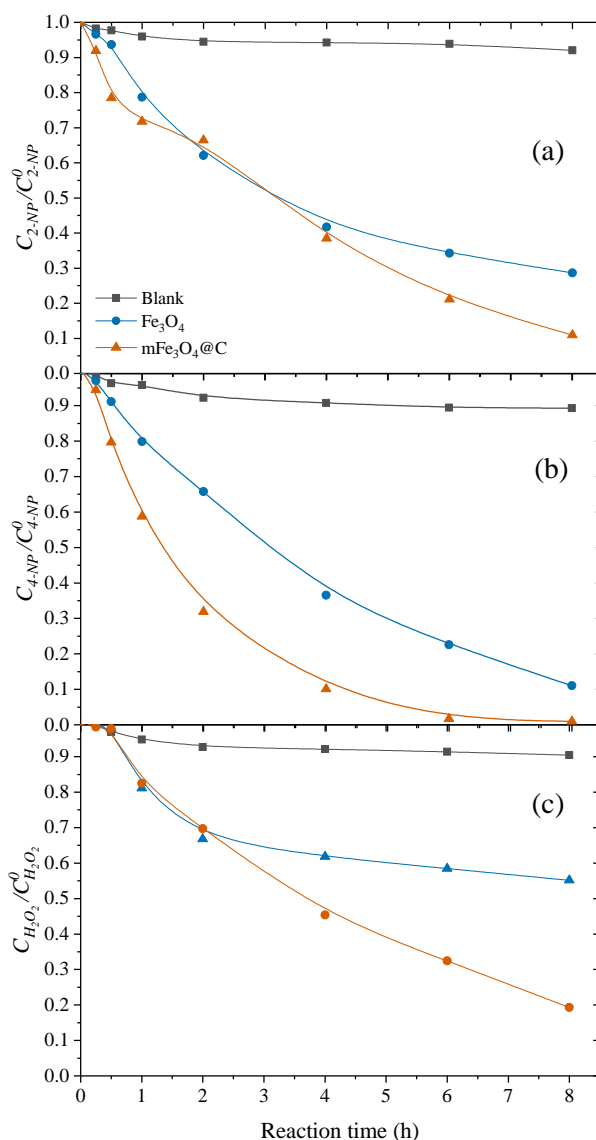


Figure 4. Concentrations of (a) 2-NP, (b) 4-NP, and (c) H<sub>2</sub>O<sub>2</sub>, throughout oxidation reactions. Lines are only used to guide reading the removal trends. Operating conditions: 80 °C, pH 3.5,  $C_{o-NP}^0 = C_{p-NP}^0 = 50 \text{ mg}\cdot\text{L}^{-1}$ ,  $C_{H_2O_2}^0 = 696 \text{ mg}\cdot\text{L}^{-1}$ ,  $C_{cat} = 2.5 \text{ g}\cdot\text{L}^{-1}$ .

**Conclusion.** In conclusion, the findings of this study highlight the tremendous potential of the system as a highly effective and viable alternative for the efficient cleansing of contaminated streams polluted with 2-nitrophenol and 4-nitrophenol. By combining mFe<sub>3</sub>O<sub>4</sub>@C catalysts with oxidation reactions, the system demonstrated remarkable capabilities in removing pollutants from the environment. The significant enhancement in pollutant removal observed through the utilization of mFe<sub>3</sub>O<sub>4</sub>@C catalysts underscores their effectiveness as a powerful tool in environmental remediation. The carbon-coated catalyst exhibited superior performance compared to the bare core catalyst, leading to notable increases in the removal of 2-NP and 4-NP. Moreover, the decomposition of H<sub>2</sub>O<sub>2</sub> was significantly enhanced, indicating the system's potential in efficiently breaking down hazardous compounds. These results lay a solid foundation for future exploration and

development of the system for a wide range of environmental remediation applications. The remarkable pollutant removal capabilities of  $m\text{Fe}_3\text{O}_4@\text{C}$  catalysts in combination with oxidation reactions pave the way for the implementation of this approach in real-world scenarios. Continued research and development in this area hold immense promise for addressing the challenges posed by contaminated streams and promoting sustainable and effective solutions for environmental cleanup.

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#### **ЛАСТАНҒАН АҒЫНДАРДАН НИТРОФЕНОЛДАРДЫ ТИІМДІ ЖОЮ ҮШІН КӨП ЯДРОЛЫ ҚАБЫҚШАЛЫ НАНОБӨЛШЕКТЕР**

**Аңдатпа.** Бұл зерттеудің мақсаты ластанған ағынды сулардан арнайы нитрофенолдарды (2 және 4-нитрофенол) тиімді жою үшін Фентонның гетерогенді процестерінде көп ядролы қабықшалы нанобөлшектерді қолдануды зерттеу болып табылады. Суперпарамагниттік магнетиттің (Fe<sub>3</sub>O<sub>4</sub>) синтезі лимон қышқылы мен темір нитратының ерітіндісін қолдана отырып, ерітіндіні жағу әдісімен (SCS) синтезделді. MFe<sub>3</sub>O<sub>4</sub>@C катализаторы бейімделген Штебер техникасы арқылы алынды. Материал көп ядролы қабықтың қажетті архитектурасын қалыптастыруға байланысты нақты химиялық құрамдарды анықтау үшін сипатталған. Көміртегімен қапталған Катализатор өзегі жоқ катализатормен салыстырғанда жоғары өнімділікті көрсетті, бұл 2-NP және 4-NP жойылуының айтарлықтай өсуіне әкелді. Сонымен қатар, H<sub>2</sub>O<sub>2</sub> ыдырауы айтарлықтай күшейтілді, бұл қауіпті қосылыстардың тиімді бөлінуіндегі жүйенің әлеуетін көрсетеді. Бұл зерттеудің нәтижелері 2-нитрофенол және 4 нитрофенолмен ластанған ластанған су ағындарын тиімді тазартудың жоғары тиімді және өміршең баламасы ретінде жүйенің орасан зор әлеуетін көрсетеді.

**Тірек сөздер:** көп ядролы қабық, нанобөлшектер, каталитикалық ылғалды асқын тотығу.



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#### НАНОЧАСТИЦЫ С МНОГОЯДЕРНОЙ ОБОЛОЧКОЙ ДЛЯ ЭФФЕКТИВНОГО УДАЛЕНИЯ НИТРОФЕНОЛОВ ИЗ ЗАГРЯЗНЕННЫХ ПОТОКОВ

**Аннотация.** Целью данного исследования является изучение применения наночастиц с многоядерной оболочкой в гетерогенных процессах Фентона для эффективного удаления специфических нитрофенолов (2 и 4-нитрофенола) из загрязненных сточных вод. Синтез суперпарамагнитного магнетита ( $Fe_3O_4$ ) был осуществлен методом синтеза методом сжигания раствора (SCS) с использованием раствора лимонной кислоты и нитрата железа. Катализатор  $mFe_3O_4@C$  был получен с помощью адаптированной методики Штебера. Материал был охарактеризован для определения конкретных химических составов, связанных с формированием желаемой архитектуры многоядерной оболочки. Катализатор с углеродным покрытием демонстрировал более высокие эксплуатационные характеристики по сравнению с катализатором без сердцевин, что привело к заметному увеличению удаления 2-NP и 4-NP. Более того, разложение  $H_2O_2$  было значительно усилено, что указывает на потенциал системы в эффективном расщеплении опасных соединений. Результаты этого исследования подчеркивают огромный потенциал системы как высокоэффективной и жизнеспособной альтернативы для эффективной очистки загрязненных водотоков, загрязненных 2-нитрофенолом и 4-нитрофенолом.

**Ключевые слова:** многоядерная оболочка, наночастицы, каталитическое влажное пероксидное окисление.