

Fabrication of Silver-Silica Composite using the Carbo-thermal Degradation of Oil Palm Leaves for the Reduction of *p*-nitrophenol

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Abstract. Oil palm leaves are natural sources of bio-silica. In situ carbothermal degradation was conducted at 600°C using silver nitrate as a metallic silver (Ag) precursor and oil palm leaves as a silica (SiO₂) source. X-ray diffraction of the solid product (Ag@OPLA600) revealed the presence of Ag and SiO₂ from the oil palm leaves. Fourier transform infrared spectroscopy showed an absorbance consistent with the presence of SiO₂. Scanning electron microscopy revealed a solid surface with cavities. Ag@OPLA600 was used as a heterogeneous mediator to reduce *p*-nitrophenol to *p*-aminophenol within 15 minutes. This work provides a new approach for the utilization of sustainable natural resources as a metal Ag supporting material to produce a mediator for the conversion of *p*-nitrophenol to *p*-aminophenol in a simple manner.

Keywords: Carbo-thermal degradation; Heterogeneous mediator; Oil palm leaves; Silver-based composite

1. Introduction

Silica (SiO₂) is the most abundant compound in nature and is found in various living organisms. The most common sources of natural silica in living organisms are sugarcane bagasse and rice husks (Permatasari, Suchaya, and Nandiyanto, 2016; Prasad and Pandey, 2012). The high demand for silica in chemistry and chemical technology has prompted intensive research efforts toward the utilization of biogenic silica as main or supporting material (Prabha *et al.*, 2020; Bahadur *et al.*, 2011). For example, silica is commonly used as a supporting material to prepare heterogeneous catalysts (Corro *et al.*, 2017; Adam, Ahmed, and Min, 2008). Recently, a heterogeneous silver-based catalyst was prepared by simply impregnating waste rice husk with silver ions, which were converted to supported nanoparticles through carbothermal reduction under a nitrogen atmosphere (Unglaube, Kreyenschulte, and Mejía, 2021). Silver-silica derivatives (Ag₂O-SiO₂) were demonstrated to exhibit good activity toward bacteria (Hou *et al.*, 2018; Peszke *et al.*, 2017;

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Quang *et al.*, 2011), as virucidal materials (Assis *et al.*, 2021), as rice antibacterial agents (Cui *et al.*, 2016), and other developments to obtain Ag/SiO₂ with specific characteristics (Flores *et al.*, 2008; Jiang, Chen, and Mao, 2008).

The utilization of biomass from agricultural wastes to produce various materials with diverse applications has spawned considerable research (Kartohardjono, Pamitran, and Putra, 2019). In Indonesia, palm oil stands out as a popular agricultural commodity, whose production generates various types of waste. Consequently, the valorization of such wastes in, for example, the delignification of oil palm empty fruit bunches (Hermansyah *et al.*, 2019) and the extraction of palmitic from oil palm (Mulia *et al.*, 2018) is attracting increasing research attention.

In this context, oil palm leaves (OPL) are particularly interesting owing to their high silica content, which can be produced from burning OPL. Recent studies have demonstrated that OPL can be used to obtain significant amounts of silica to prepare diverse materials (Onoja *et al.*, 2018; Onoja *et al.*, 2017). For example, the authors' research group reported the use of OPL to produce silica-containing materials, which were then used as precursors in the manufacture of calcium silicate derivatives (Yudha *et al.*, 2020a), ZnO–SiO₂, and Zn₂SiO₄ (Yudha *et al.*, 2020b). Moreover, the decomposition product of OPL was used as a support material for gold particles as catalysts for the reduction of nitroarene compounds (Yudha *et al.*, 2021).

In this study, a silver-based composite (Ag@OPLA600) was fabricated via a facile and straightforward method involving a simple impregnation of silver ions into OPL ash powders followed by carbothermal degradation at 600°C to destroy the organic materials and reduce silver ions to metallic silver. The obtained composite was then used as a mediator for the heterogeneous reduction of *p*-nitrophenol (*p*-NP) to *p*-aminophenol (*p*-AP), which is one of the intermediates in the synthesis of *N*-(4-hydroxyphenyl) acetamide (commonly known as paracetamol) via a simple acetylation process (Abdullaev *et al.*, 2014). Heterogeneous systems are often used as reaction mediators or catalysts due to their easy separation, recyclability, and reusability. In this case, silver particles as the active material are either attached to the surface or trapped in silica for the reduction of *p*-NP to *p*-AP.

2. Experimental Methods

2.1. Materials

All glassware and crucibles used in the experiments should be cleaned with a freshly prepared aqua-regia solution. The chemical reagents such as silver nitrate, acetone, and *p*-nitrophenol were purchased from Merck (Germany), and NaBH₄ was purchased from Sigma–Aldrich (Singapore); demineralized water was purchased from Brataco Chemika (Indonesia). The chemicals used in this work were of analytical grade and were used as received without any further purification. The OPL powder preparation follows the previously reported procedure (Yudha *et al.*, 2020a).

2.2. Methods

The Ag@OPLA600 composite was synthesized using a solid-state reaction between OPL and silver nitrate (AgNO₃) according to a modified literature procedure (Unglaube, Kreyenschulte, and Mejía, 2021). Specifically, nitrogen gas was not used in this study for the solid-state reaction. Briefly, 1 g of AgNO₃ was placed in a solvent mixture of acetone/demineralized water (50 mL/5 mL) in a crucible and stirred until completely dissolved. Subsequently, 4 g of OPL powder was added to the AgNO₃ solution with stirring until all the OPL powder was suspended. The reaction mixture was heated to 70°C to form a gel on the bottom of the crucible. The gel was left to stand for 12 hours and then placed in a furnace, where the temperature was increased to 600°C for 2 hours and maintained at

600°C for another 3 hours before cooling down for 12 hours. The as-obtained Ag@OPLA600 composite was characterized via X-ray diffraction (XRD MiniFlex, Rigaku) and Fourier transform infrared (FTIR, Compact FT-IR Alpha 2, Bruker) spectroscopy to obtain the phase and vibration patterns, respectively, and scanning electron microscopy (SEM, JEOL, JED-2300) and energy-dispersive X-ray spectroscopy (EDX) for the surface and elemental analysis, respectively.

The catalytic performance of Ag@OPLA600 was evaluated in the reduction of nitroarene using a slightly modified reported procedure (Kadam and Tilve, 2015). Ag@OPLA600 (50 mg) was added to a reaction mixture of *p*-NP (1 mL, 2 mM), NaBH₄ (5 mL, 30 mM), and demineralized water (10 mL) with gentle stirring. The UV-Visible spectra of the solution were recorded over the range of 200–600 nm using an Agilent-60 spectrophotometer. The reaction progress was monitored every 5 minutes until the nitrophenolate ion intermediate completely disappeared. The solution was picked up using dropper pipettes directly without centrifugation for UV-Visible measurement and returned after analysis. The synthesis of the as-prepared materials and the reduction of *p*-NP to *p*-AP are illustrated in Figure 1.

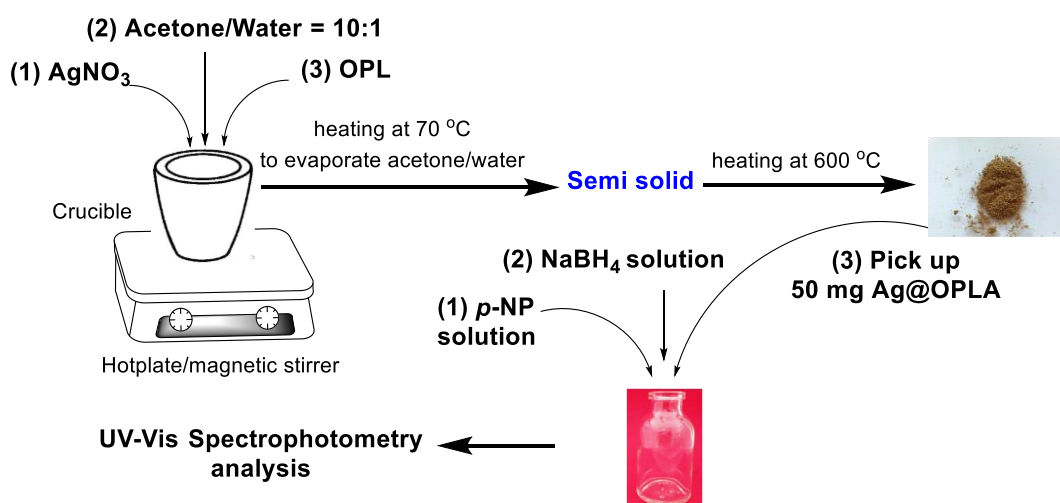


Figure 1 Illustration/scheme for the experimental procedure in preparing composite and its function in reduction of *p*-nitrophenol (*p*-NP) to *p*-aminophenol (*p*-AP)

3. Results and Discussion

3.1. Composite Preparation and Characterization

The carbothermal degradation process afforded a grey-white powder. Its XRD pattern showed a broad peak at $2\theta = 22^\circ$ and several sharp peaks at 38.2° , 44.3° , 64.4° , and 77.4° (Figure 2a). This is in line with the JCPDS card number 85-0335 for amorphous silica (Musić, Filipović-Vinceković, and Sekovanić, 2011) and JCPDS card number JCPDS 04-0783 for metallic silver (Ag (0)) (Syed et al., 2019; Yang, Zhang, and Huang, 2013; Park et al., 2011). Heating AgNO₃ at low temperatures resulted in combustion with oxygen to form silver (I, III) oxide (AgO) and silver(I) oxide (Ag₂O). In contrast, heating above 400°C led to cleavage of the Ag–O bond, eventually producing Ag metal as the decomposition product (Waterhouse, Bowmaker, and Metson, 2011). The reaction did not require the presence of inert gases such as nitrogen and argon. The XRD pattern showed peaks corresponding to OPL powder, which exhibits amorphous peaks, except two broad peaks at $2\theta = 21.6^\circ$ and 29.9° (Yudha et al., 2020a). The XRD analysis revealed the disappearance of the peaks of the starting materials and the appearance of new peaks of the composite material.

Figure 2b shows peaks at 3418 cm^{-1} (OH stretching), 2922 cm^{-1} (C–H stretching), 1632 cm^{-1} (–O–tensile vibration), and 1040 cm^{-1} (C–OH bending). Figure 2c shows the FTIR spectra of the Ag@OPLA600 solid. The peaks at 3430 and 1634 cm^{-1} can be assigned to the stretching and bending vibration of the OH group from water adsorbed on the surface of the solid silica material. The sharp peak at 1094 cm^{-1} and the small peak at 799 cm^{-1} can be attributed to the Si–O–Si stretching vibration (Deshmukh, Peshwe, and Pathak, 2012). Although the characteristic peaks of the Ag–O–Si bond were not observed in the FTIR spectrum, the XRD result (Figure 2a) confirmed that Ag@OPLA600 contained Ag metal. Taken together, the XRD and FTIR spectroscopy results confirmed the successful synthesis of the composite.

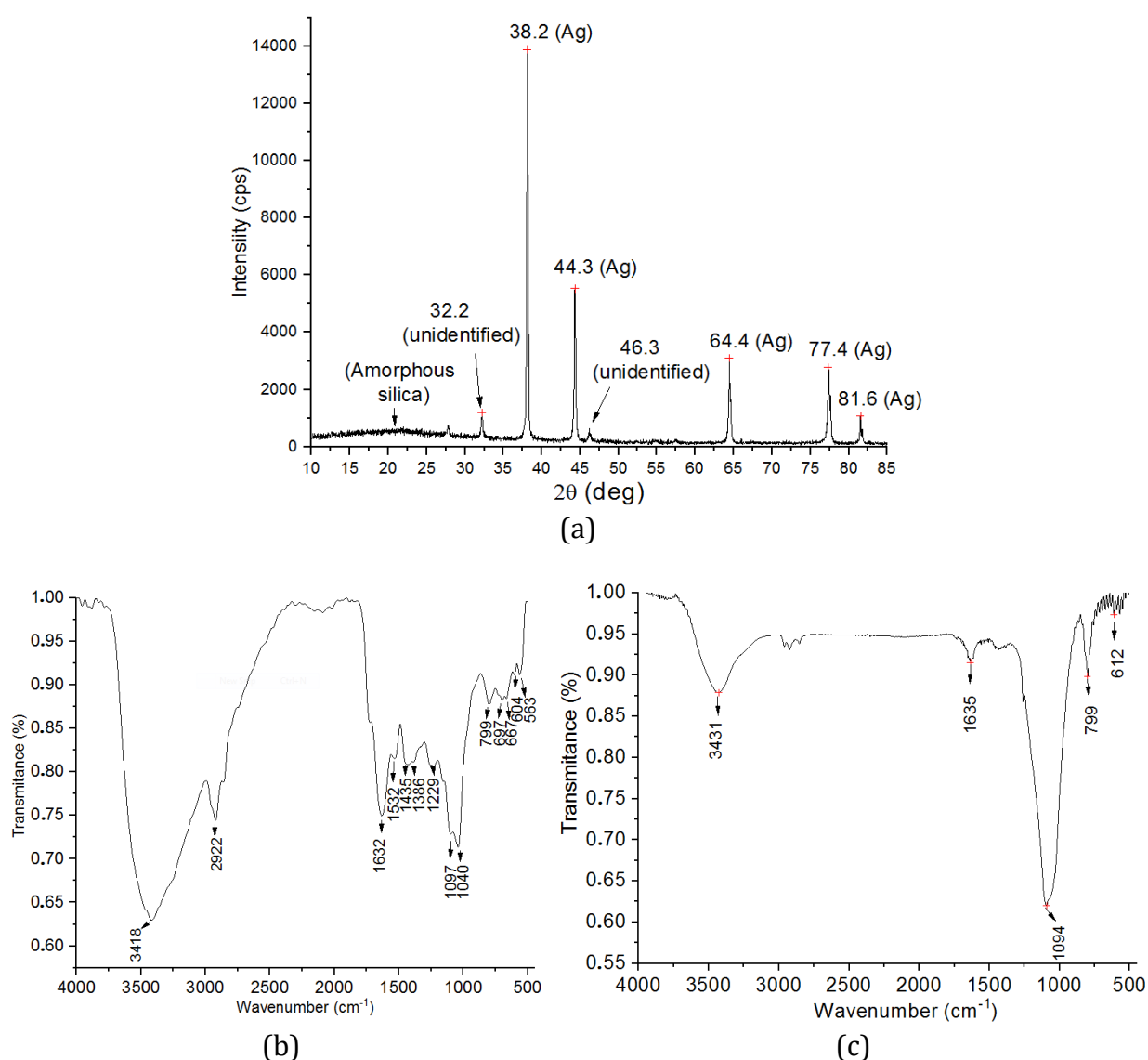


Figure 2 (a) XRD pattern of Ag@OPLA600, (b) FTIR spectrum of OPL powder (c) FTIR spectrum of Ag@OPLA600

The SEM image (Figure 3) showed that the surface of the Ag@OPLA600 composite was irregular and contained large pores. Small grains due to Ag metal were also observed. Table 1 summarizes the results of the EDX analysis, which demonstrated that the composite contained Si, Ag, and some impurities in very small quantities, such as K, Ca, Mg, and Cu. The amount of Ag in the as-prepared materials was 19.73%, which was smaller than in the original precursor (63.5%) owing to the presence of other elements in Ag@OPLA600. The

presence of other materials that could support metallic Ag, especially silica, was thereby demonstrated.

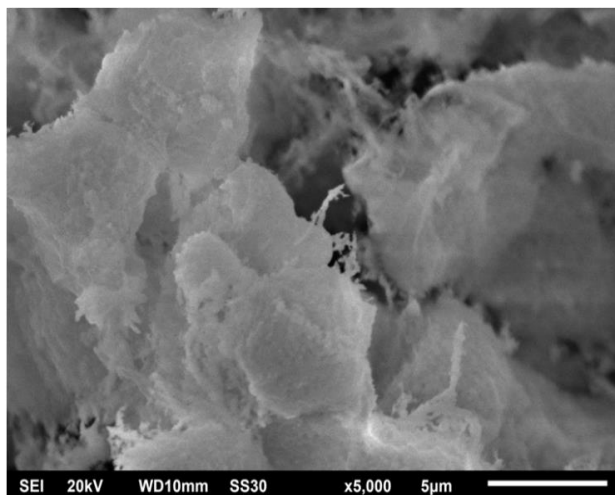


Table 1 EDX analysis result of the as prepared Ag@OPLA600

Element	K(eV)	% Mass
C	0.277	5.12
O	0.525	49.1
Mg	1.253	0.42
Si	1.739	19.45
P	2.013	0.40
Cl	2.621	0.39
Ag	2.983	19.73
K	3.312	2.08
Ca	3.690	2.05
Cu	8.040	0.87
Zn	8.630	0.48

Figure 3 SEM-images of Ag@OPLA600 (magnification 5000×)

3.2. Composite Application

The Ag@OPLA600 composite was applied as a heterogeneous mediator for the reduction of *p*-NP with the assistance of hydrogen produced in situ from NaBH₄ dissolved in water. The reaction could be followed with the naked eye as follows: When NaBH₄ was added to the solution of *p*-NP, the color changed from transparent to yellow due to the formation of a *p*-nitrophenolate intermediate. Interestingly, the yellow solution slowly turned transparent again when Ag@OPLA600 was added due to the conversion of *p*-nitrophenolate to *p*-AP, as shown in Figure 4.

A UV-Visible spectrophotometry analysis was performed to gain more insight into the transformation of *p*-NP to *p*-AP. Figure 4a displays a spectrum of *p*-NP in water, which shows a clear absorbance at 316 nm. The solution of this compound was clear. Then, the addition of NaBH₄ to the *p*-NP solution resulted in a deprotonation reaction and the formation of nitrophenolate ions (yellow solution), as indicated by a new peak at 401 nm (Figure 4b) (Nurwahid *et al.*, 2022; Hou *et al.*, 2016). The Ag@OPLA600 composite helped deliver hydrogen ions from NaBH₄ to the nitro group in the *p*-nitrophenolate ion structure. The reduction of the nitro group to the amine group in the presence of Ag@OPLA600 occurred within 15 minutes, evidenced by the rapid disappearance of the *p*-NP peak. The disappearance of the peak of the 4-nitrophenolate ions was accompanied by a new peak at 301 nm due to *p*-AP (Figure 4c).

The reaction of nitrophenolate ions is known to be kinetically hindered and affected by the repulsion between BH₄⁻ and *p*-nitrophenolate ions; therefore, it does not occur in the absence of a suitable catalyst (Kalantari *et al.*, 2019; Zhang *et al.*, 2018; Baruah *et al.*, 2013; Li and Chen, 2013; Huang *et al.*, 2010). To gain more insight into this reaction, a qualitative analysis was performed using pristine *p*-NP and *p*-NP treated with Ag@OPLA600 in the absence of NaBH₄, as shown in Figure 5. *p*-nitrophenol (*p*-NP) (Figure 5a) was converted to *p*-nitrophenolate ions (Figure 5b) in the presence of Ag@OPLA600, which is sufficiently basic to promote the ionization of the phenol group in a water environment. However, due to the lack of a hydrogen source in the reaction mixture, the conversion to *p*-AP did not proceed even when the reaction mixture was left to stand at room temperature for 24 h.

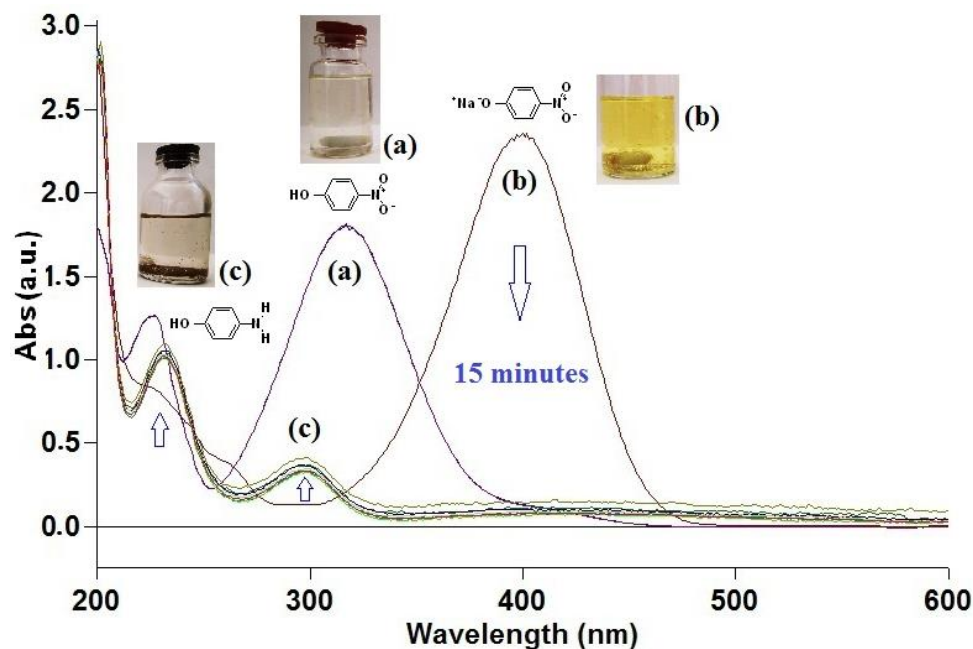


Figure 4 UV-Visible Spectrophotometry analysis of *p*-nitrophenol reduction in the presence of NaBH_4 and Ag@OPLA600 ; (a) *p*-nitrophenol spectrum, (b) *p*-nitrophenolate ion spectrum, (c) *p*-aminophenol spectrum

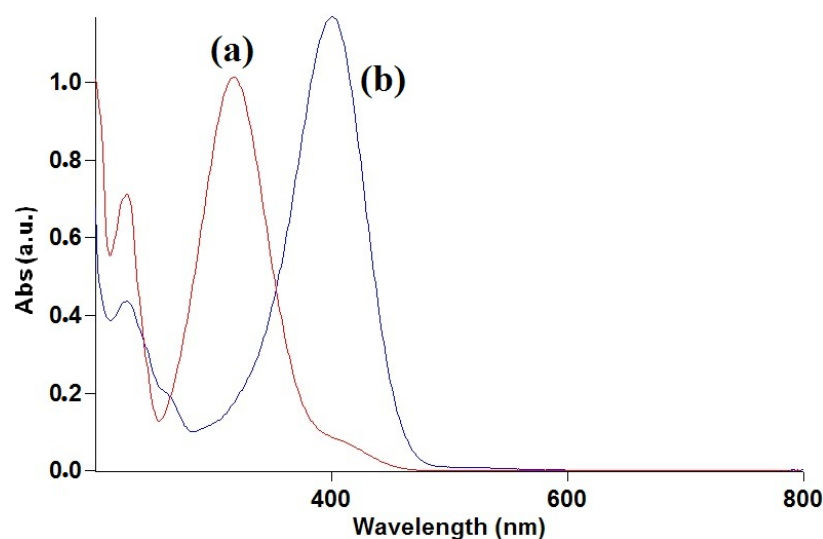


Figure 5 UV-Visible Spectrophotometry analysis of *p*-nitrophenol reactivity in the presence of Ag@OPLA600 ; (a) *p*-nitrophenol spectrum, (b) *p*-nitrophenolate ion spectrum

According to the results depicted in Figures 4 and 5, the mechanism shown in Figure 6 was proposed. The reduction of *p*-NP to *p*-AP is catalyzed by Ag nanoparticles or supported Ag particles, as previously reported (Al-Namil, Khoury, and Patra, 2019; Rajamanikandan, Shanmugaraj, and Ilanchelian, 2017). In particular, an isotope-labeling study revealed that water (not NaBH_4) is the source of hydrogen and that the reaction systems are stabilized by BH_4^- (Kong et al., 2017). Ag@OPLA600 appears at the bottom of the vial after the reaction is complete, which indicates that the reaction is heterogeneous. The solid material can be easily separated by slowly decanting the solution after the reaction.

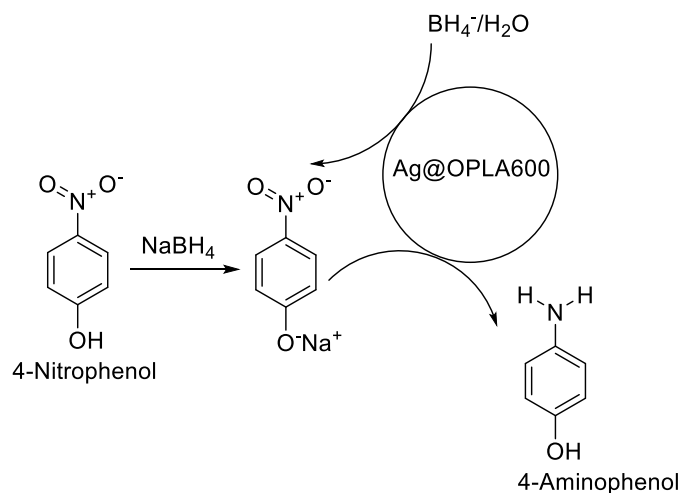


Figure 6 The possible general reaction mechanism of reduction of the *p*-nitrophenol to *p*-aminophenol in the presence of Ag@OPLA600

This carbothermal degradation of OPL in the presence of AgNO_3 is advantageous regarding eco-friendliness and cost-effectiveness. First, a cost-effective fabrication is achieved using a one-pot process to form Ag@OPL600 . Second, nontoxic and sustainable raw materials are used, and the generation of chemical waste is minimized.

4. Conclusions

A green solid–solid reaction between OPL and AgNO_3 at 600 °C produced a solid Ag@OPLA600 composite, which was used as an inexpensive mediator for the reduction of *p*-NP to *p*-AP. The results demonstrate the potential of this approach for waste and biomass valorization, especially in the case of palm oil biomass. These findings could pave the way for the development of sustainable and cost-effective technologies for reducing waste and utilizing renewable resources. While further research is needed to fully explore the potential of this approach, the results of this study represent an important step forward in this field.

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