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HIGHLY EFFICIENT ONION PEEL ASH CATALYST FOR DEGRADATION OF METHYLENE BLUE

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Abstract: Methylene blue is a dye commonly used by the textile industry. However, its presence in textile effluent poses significant risks to human health and water resources. Various environmental technologies have been developed to remove this refractory pollutant from contaminated effluent. However, those techniques have limitations, such as high operational costs and use of unsustainable resources. In this study, the efficacy of an ash catalyst derived from household waste, namely onion peel waste, for degrading methylene blue was evaluated. Inductively coupled plasma-optical emission spectroscopy analysis revealed the presence of magnesium oxide, MgO, (528.00 ± 40.00 ppb) as one of the primary elements in onion peel ash, crucial for methylene blue degradation. Additionally, Fourier-transform infrared spectroscopy analysis identified an absorption band at around 871 cm^{-1}, indicating the presence of MgO in the onion peel ash. The findings revealed that onion peel ash degraded methylene blue with an 84% (0.00616 min^{-1}) degradation rate under sunlight and a 95% (0.00578 min^{-1}) degradation rate under UV-lamp exposure. This research suggests that onion peel ash holds promise for effectively degrading methylene blue in textile effluents, offering a sustainable solution for addressing environmental concerns associated with textile dye pollution.

Keywords: Degradation, methylene blue, green chemistry, onion peel ash, biowaste.

Introduction

Dyes are a class of organic compounds widely used as colouring agents in a variety of commercial industries, including textiles, paints, leather, cosmetics, food, pharmaceutical, and pigment manufacturing (Ba *et al*., 2020; Dahiya & Nigam, 2020; Mohammed *et al*., 2020; Vasiljevic *et al*., 2020; Hanafi & Sapawe, 2021; Slama *et al*., 2021; Mohan & Prasad, 2022). It is estimated that there are more than 10,000 distinct dyes and pigments in industrial use and more than 700,000 tonnes of synthetic dyes are produced annually on a global scale (Maria *et al*., 2013; Agustiono *et al*., 2020). However, the discharge of dye effluents has a significant impact on the environment, and most of them are highly toxic and non-biodegradable (Sree *et al*., 2020; Vasiljevic *et al*., 2020). The textile industry utilises a substantial quantity of water in its production processes, and up to 200,000 tonnes of dyes are lost to effluents annually during dyeing and refining processes (Blinová, 2019). Another study estimates that approximately 280,000 tonnes of textile dyes are discharged annually into dye-containing industrial wastewater (Gita *et al*., 2019). Unfortunately, due to their high resistance to light, water, temperature, detergents, soap, chemicals, bleach, and perspiration, most dyes evade conventional wastewater treatment processes and persist in the environment (Maria *et al*., 2013; Blinová, 2019). This persistence leads to water pollution, which poses a significant threat to aquatic life and the ecosystem as a whole (Mardikar *et al*., 2020).

Among numerous dyes, methylene blue is frequently found in textile wastewater (Agustiono *et al*., 2020). Methylene blue is an intensely coloured compound that is commonly used in the dyeing and printing of textiles, and it is considered highly toxic and carcinogenic (Rauf *et al*., 2010; Vasiljevic *et al*., 2020). Adsorption, membrane filtration, ion exchange, coagulation, electrochemical treatment, integrated Fenton's oxidation, sedimentation sonochemical degradation, and biological treatment are some of the common methods used to remediate organic pollutants such as methylene blue and its degradation byproducts (Maria *et al*., 2013; Tichapondwa *et al*., 2020). Although these technologies have been demonstrated to be effective, they all suffer from incomplete treatment or elimination as the dye pollutant is merely being transferred from one medium to another (Tichapondwa *et al*., 2020). In addition, these techniques have disadvantages, one of which is the high operational cost resulting from their extensive energy consumption (Agustiono *et al*., 2020). As such, developing a more environmentally friendly, cost-effective, and energy-efficient strategy for dye degradation is vital for removing these toxic organic compounds from ecosystems.

Photodegradation is an advanced oxidation process that is extensively used for methylene blue elimination (Khan *et al*., 2022). It has the advantage of breaking down chemically complex molecules into simple, nontoxic, and lower molecular weight fragments due to light exposure (Saeed & Khan, 2017). This is an emerging and promising technology for wastewater treatment with the capability of decolourising and degrading the dye molecules into simple and innocuous inorganic compounds, such as CO_2 and H_2O (Khan *et al*., 2019). However, due to the stability of methylene blue under visible light irradiation, photolysis and catalysis alone cannot degrade it effectively (Khan *et al*., 2022). Nanomaterials, such as zinc oxide (ZnO), titanium dioxide (TiO_2), zinc sulphide (ZnS), iron titanate (Fe_2TiO_5), and magnesium oxide (MgO), were efficiently utilised as catalysts in methylene blue dye degradation (Balcha *et al*., 2016; Mani *et al*., 2018; Anil *et al*., 2019; Balakrishnan *et al*., 2020; Tichapondwa *et al*., 2020; Vasiljevic *et al*., 2020). Unfortunately, the overexploitation of certain metals for industrialisation has led to element depletion in the coming century, a phenomenon known as resource deficit (Hunt *et al*., 2015). These endangered elements can be replaced by alternative metal oxides, particularly those derived from biowaste which exhibit superior catalytic performances (Sree *et al*., 2020).

The onion (*Allium cepa* L.) is the second most important horticultural crop after tomato, prized for its culinary versatility and wide-ranging applications (Pareek *et al*., 2017). It is reported that 99,968,016 tonnes of onions are produced from an area of 5,192,651 hectares worldwide (Sagar *et al*., 2021). However, up to 25% of qualitative and quantitative postharvest losses occur during storage. Additionally, the increased processing of onions has also generated enormous processing waste, especially onion peels. Reports indicate that within the European Union alone, the onion processing sector discards more than 500,000 tonnes of onion waste every year, mostly from the United Kingdom, Spain, and the Netherlands (Sharma *et al*., 2016). Its peculiar aroma renders it unsuitable for extensive use as animal feed (Celano *et al*., 2021; Sagar *et al*., 2021). Without appropriate treatment, this biowaste could negatively impact the environment and living organisms (Chia *et al*., 2019).

As part of a continuing exploration of sustainable chemistry, herein, this study examines the transformation of waste onion peel into onion peel ash catalyst via direct burning in a furnace for use in the degradation of methylene blue. Chia *et al.* (2019) have previously investigated the use of onion peel and its chemical composition for organic transformations. It is hypothesised that MgO nanoparticles present in onion peel ash could catalyse the degradation of methylene blue. This onion peel ash underwent characterisation using inductively coupled plasma-optical emission spectroscopy (ICP-OES) and scanning electron microscope (SEM) to determine its physical and chemical properties. The catalyst's activity was then tested for its ability to degrade methylene blue dye solutions under both sunlight and UV-lamp exposure. The degradation percentage of methylene blue was determined using UV-visible spectroscopy. This study is significant and differs from the previous works made on dye degradation or removal (Usha *et al.*, 2020; Abid *et al.*, 2021; Aswathi *et al.*, 2021) by presenting a highly efficient and straightforward method of preparing ash onion peel ash catalyst without the relying on external metals for environmental protection purposes.

Materials and Methods

Chemicals and Reagents

In the current study, chemical reagents and solvents, including methylene blue dye, nitric acid, and hydrochloric acid, were purchased from Sigma Aldrich (Malaysia). Fourier-transform infrared spectroscopy (FT-IR) analysis was performed using a Perkin Elmer 400 FT-IR instrument outfitted with a GladiATR attenuated total reflectance accessory. The measurement was conducted over the range of 4,000-400 cm^{-1} at a resolution of 8 cm^{-1} and 40 scans per sample.

Preparation of the Onion Peel Ash

Onions were obtained from a nearby local supermarket. The outer layer and bulb of the onion were separated. The onion peels were then sliced into small pieces and air-dried for three days. The onion peel ash was prepared according to the method by Chia *et al*. (2019). In brief, the dried peels (130 g) were burnt in the furnace at 500˚C for about 1 hour. The resulting ash was then transferred into a sample vial and stored for subsequent use.

Characterisation of Onion Peel Ash Inductively Coupled Plasma-optical Emission Spectroscopy

The onion peel ash was pre-treated before being characterised using ICP-OES (Varian Vista Pro). Initially, 0.05 g of onion peel ash was mixed with 1.5 mL of nitric acid. The sample was then put in the oven for 7 hours at 115ºC in closed-air conditions. Next, 1 mL of the sample was transferred into a beaker and diluted with 9 mL of deionised water until the sample becomes clear. Then, the metal content of the onion peel ash was analysed using ICP-OES. The sample for ICP-OES was analysed in triplicates and the data were expressed in mean value ± standard deviation (SD).

Scanning Electron Microscope

The surface morphology of the onion peel ash was characterised using SEM (JEOL JSM-6360 LA). The onion peel ash was attached to round copper stubs and coated with gold to create contrast for SEM imaging, produce high-resolution images of the surfaces of the onion peel ash sample at high magnification, and shield the sample surfaces from electron beams with kinetic energies between 1 to 1.25 kV. The coated sample was then placed within the SEM instrument for further analysis. The samples were analysed using an acceleration of 10 kV with a magnification of x5,000.

The Catalytic Activity of Onion Peel Ash by Photodegradation Experiment

The catalytic activity of onion peel ash was assessed through the photodegradation of aqueous methylene blue. The photodegradation experiment was conducted following a previously established procedure with minor modifications (Sree *et al*., 2020; Chook *et al*., 2022). In short, the photodegradation of methylene blue was evaluated by exposing aqueous methylene blue to sunlight and UV-lamp with and without the addition of onion peel ash. Subsequently, a beaker containing 24 mL of methylene blue (10 ppm) was mixed with 24 mg of onion peel ash. The mixture was then stirred at 100 rpm for 15 minutes in the absence of light to attain the adsorption and desorption equilibrium between onion peel ash and aqueous methylene blue. The photodegradation of methylene blue was performed in triplicate and evaluated by measuring its absorbance at 0, 1, 2, and 3 hours of sunlight and UV-lamp (15 W) exposure at a wavelength of 665 nm using UV-visible spectroscopy (Shimadzu

Corporation, Tokyo). Equation 1 was used to calculate the percentage of methylene blue degradation (Sirajudheen & Meenakshi, 2019):

$$\% \text{ Degradation} = [(C_o - C_t)/C_o] \times 100 \quad (1)$$

where C_o is the initial concentration of methylene blue and C_t is the final concentration of methylene blue.

Results and Discussion

Characterisation of Onion Peel Ash ICP-OES Analysis

The metal content of onion peel ash was determined using ICP-OES, which produced the following output (mean value ± SD) in parts per billion (ppb): Potassium (11749 ± 227.00), magnesium (528.00 ± 40.00), sodium (92.00 ± 0.21), phosphorus (87.00 ± 5.00), yttrium (33.00 ± 0.02), and boron (32.00 ± 3.00). Based on the results, magnesium is found to be one of the primary metals in onion peel ash, which is in agreement with a prior study (Chia *et al*., 2018). According to Zheng *et al.* (2019), magnesium oxide (MgO) was employed as the catalyst for the degradation of various dyes in an aqueous solution due to its large gap, low dielectric constant, and refractive index. MgO was also found to be non-toxic, which aligns with green principles (Kumar *et al*., 2022). Thus, MgO contained in onion peel ash could potentially act as a catalyst in the degradation of methylene blue.

Scanning Electron Microscopy (SEM) Analysis

The surface morphology of onion peel ash was investigated using SEM as shown in Figure 1. The SEM image of onion peel ash shows a porous and agglomerate morphology. These pore sites present in onion peel ash could serve as catalytic sites for methylene blue degradation. The results aligned with a prior study, which suggested the high porosity of powdered onion peel could exhibit catalytic functions (Tan *et al*., 2020).

Fourier-Transform Infrared Analysis

In the FTIR spectrum of onion peel ash (Figure 2), the absorption band at around 3734 cm^{-1} corresponds to the stretching of H–O–H. This observation aligns with existing literature indicating that MgO surfaces readily absorb water molecules when exposed to the atmosphere. Likewise, the absorption band at around 871 cm^{-1} is attributed to the presence of MgO. Additionally, the absorption band at 1411 cm^{-1} can be attributed to the asymmetrical and symmetrical stretching of (O–C=O), which could be due to the presence of minor impurities

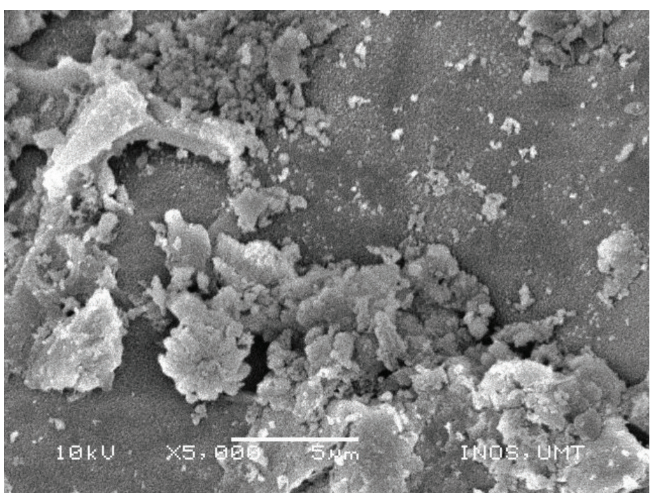


Figure 1: The SEM image of onion peel ash shows the high porosity of powdered onion peel that can exhibit catalytic functions

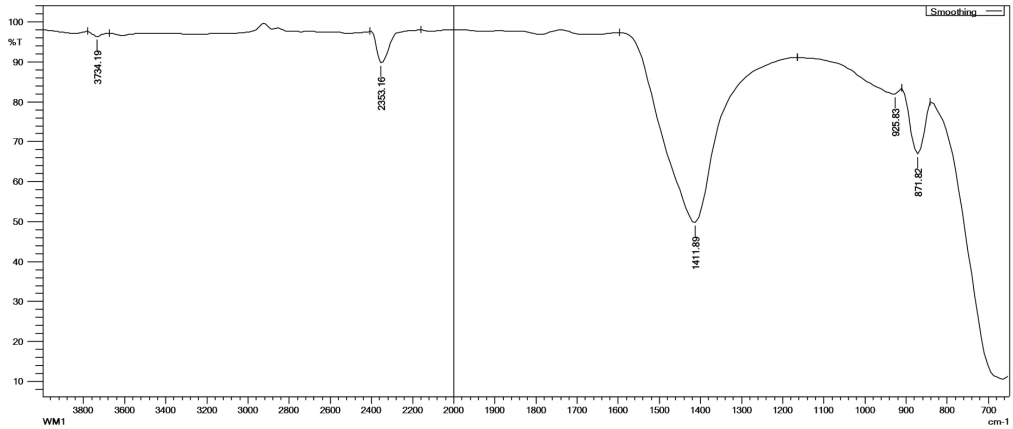


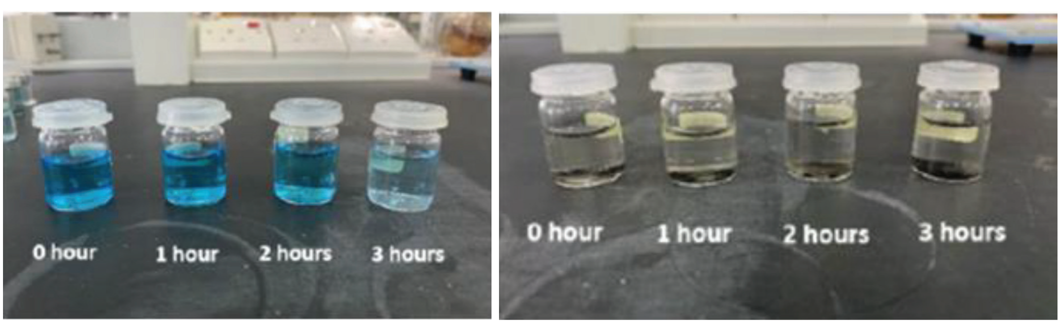
Figure 2: The FTIR spectrum of onion peel ash sample prepared via a burning process in a furnace at 600°C

in the precursors when preparing the onion peel ash. Overall, the finding agrees with previous literature that confirms the presence of MgO as the main element of onion peel ash catalyst (Zahir *et al*., 2019).

Photodegradation of Methylene Blue Catalysed by the Onion Peel Ash

The catalytic activity of onion peel ash was evaluated for the degradation of methylene blue under natural sunlight and UV-lamp exposure, and the results are shown in Figures 3 and 4. Figure 3 (a) depicts the colour changes in methylene from 0 to 3 hours in the absence of an onion peel ash catalyst, while Figure 3 (b) shows the decolourisation in methylene blue in the presence of an onion peel ash catalyst. Notably, Figure 3 (b) demonstrates a significant colour change in the methylene blue compared with Figure 3 (a), indicating that onion peel exhibits good catalytic ability in dye degradation under both sunlight and UV irradiation. These results can be further through the UV-vis analysis.

Based on the results as shown in Figure 4, methylene blue experienced only a 57% degradation after 3 hours of exposure to sunlight without the presence of a catalyst [Figure 4 (a)]. However, onion peel ash demonstrated superior catalytic activity, in which methylene blue achieved a nearly maximal degradation rate at 95% in 0 hours in the presence of sunlight and onion ash catalyst [Figure 4 (b)]. When the experiment was repeated under UV irradiation, methylene blue showed 84% degradation after 3 hours with the presence of onion ash catalyst [Figure 4 (d)]. Overall, the presence of onion peel ash has enhanced the efficiency of methylene blue degradation in both sunlight and



(a) (b)

Figure 3: Visible colour changes of methylene blue (from 0 to 3 hours) (a) in the absence and (b) presence of onion peel ash

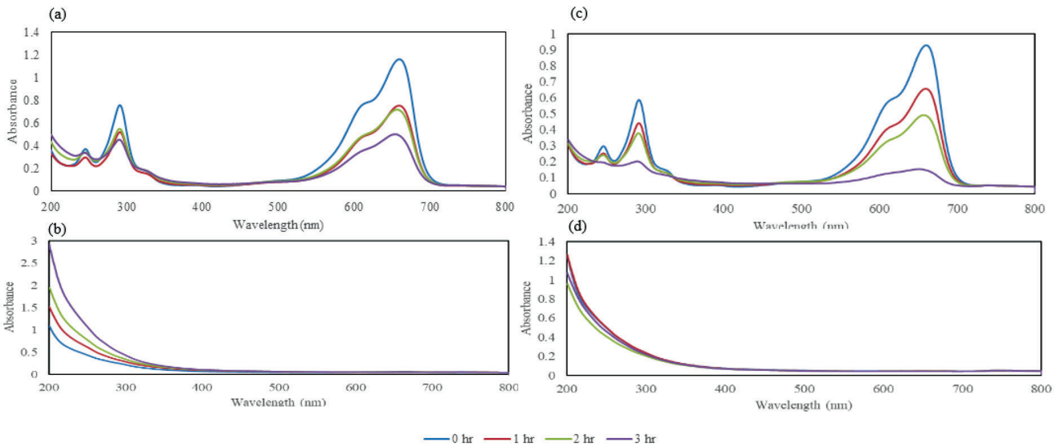


Figure 4: Degradation of methylene blue in the absence of onion peel ash under sunlight

UV irradiation experiments. This superiority is due to the presence of MgO, one of the primary elements in onion peel ash catalysts, and the result of this study is supported by recent literature (Rahmawati & Rohmawati, 2022). Therefore, onion peel ash holds promise as a potential catalyst for dye degradation and promotes the sustainable reuse of discarded onion peel ash.

Conclusions

Onion peel ash derived from waste was successfully prepared and characterised to investigate its properties. Analysis using ICP-OES revealed that magnesium was one of the most abundant metals in onion peel ash. Moreover, SEM imaging displayed the porous and agglomerate morphology of the onion peel ash. This study demonstrated that onion peel ash exhibited catalytic activity in the degradation of methylene blue, achieving 84% degradation under UV irradiation and 95% degradation under sunlight. These findings suggest that discarded onion peel ash has the potential for application in dye degradation within the textile industry.

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Conflict of Interest Statement

The authors declare that they have no conflict of interest.

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