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Removal of Organophosphate Insecticides in Runoff Water Model Consists of Fine Micro Bubble Water Generator and Absorptive Organobentonite Ceramic Composites Balls

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***Abstract*—A system for removing organophosphate insecticides was designed using modified bentonite and a fine micro-nanobubble generator. The native bentonite was modified with berberine chloride and MgO to form a berberine chloride/MgO composite powder, which was then used to fabricate ceramic porous balls approximately 2.0 cm in diameter and weighed around 7.2 g, with a permeability of approximately 1.5 × 10^6 mD. Micro-nanobubbles were generated with mean and mode sizes of 193.9 ± 13.7 nm and 107.7 ± 2.3 nm, respectively. The density of micro-nanobubbles was approximately 7×10^8/mL ± 1×10^7. To evaluate the system's effectiveness, tap water was spiked with 5 ppm of each organophosphate insecticide: methyl parathion, chlorpyrifos, profenofos, and dimethoate. The contaminated water was then passed through a filter composed of the modified bentonite ceramic balls. The concentration of each insecticide in the water tank was analyzed over time at 6, 12, 18, and 24-hour intervals using high-performance liquid chromatography. The results revealed a linear decrease in insecticide concentrations over time whereas chlorpyrifos was reduced at 15%, 32%, 40%, and 42%, methyl parathion was at 10%, 25%, 30%, and 38%, profenofos was at 18%, 35%, 40%, and 45% and dimethoate was at 8%, 20%, 30%, and 30% after 6, 12, 18, and 24 hours, respectively. The hybrid system demonstrated an ability to absorb organophosphate insecticides, suggesting its potential application for reducing insecticide residues in contaminated water zones, particularly in agricultural areas.**

***Keywords*—organophosphate insecticide, modified bentonite, micro nanobubbles water, degradation, ceramic ball, run-off water**

I. INTRODUCTION

Southeast Asia is currently experiencing significant contamination of water zones due to agricultural chemicals, as reported in investigative studies [1, 2] and frequently highlighted in news reports. The use of pesticides and insecticides in agricultural practices has led to significant environmental concerns regarding the pollution of water sources and these contaminants bring risks to both aquatic ecosystems and human health daily. Clean-up techniques using activated carbon [3], activated sludge [4], and chemical water treatment by Fe_3O_4-based absorbents [5] have been developed and applied for water treatment in past decades. However, the conventional methods have limitations such as cost, sustainability and long-term application.

The efforts to develop environmentally friendly insecticides, e.g. flavocides [6, 7], and smart agricultural technologies have been expanding. These strategies involve the application of artificial intelligence (AI), automation, and the Internet of Things (IoT) to manage and control crops [8–13]. Although these innovations contribute to reducing the dependence on chemical pesticides but their contamination remains still requiring additional remediation strategies.

The development of new insecticide-absorptive materials and novel methods for on-field for the removal of insecticides from contaminated water have been exploring. Among them, berberine-modified bentonite clay has demonstrated high absorptivity toward organophosphate insecticides, including methyl parathion, profenofos, chlorpyrifos, and dimethoate, which are widely used in Southeast Asia [14–17]. These organophosphate insecticides are known to accumulate in the environment and long-term damage the surrounding ecology. Taking into this consideration, we developed a novel berberine-modified bentonite clay-based absorptive material and used it to fabricate ceramic balls with approx. 2 cm in diameter. These ceramic balls were integrated into a run-off water system equipped with a fine micro-nano bubble water generator. The integration of micro-nano bubbles enhances the interaction between the ceramic adsorbent and insecticide-spiked water, thereby increasing the efficiency of insecticide removal. Additionally, the micro nano bubbles improve oxygenation and physical breaking down of the tested OP insecticides with aim to further improve the water quality.

The objective of this study is to evaluate the effect of our system in removing tested organophosphate insecticides. We aim to determine the adsorption capacity, efficiency, and overall feasibility of the system for on-site applications. This study also investigates the stability and reusability of the ceramic balls, assessing their long-term performance in run-off water treatment applications. The combined properties of berberine-modified bentonite clay and micro-nano bubble technology might contribute to water treatment solutions.

Here, we describe the development and optimization of the system, including the synthesis of berberine-based bentonite ceramic balls, the design of the closed run-off water treatment system, and the characterization of its impact on organophosphate (OP) insecticides. Furthermore, we examine the potential scalability of this system and its broader implications for mitigating pesticide pollution in agricultural areas. We also discuss potential policy implications and the feasibility of integrating this system into existing agricultural water management frameworks. Through this work, we expect to provide a practical and

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innovative approach to improving water quality and promoting environmental sustainability in Southeast Asia and beyond.

II. LITERATURE REVIEW

Removal technologies for organophosphate insecticides have been widely studied, particularly in Asia, China, and Africa. Various methods have been explored to eliminate these contaminants from water, soil, and food sources. For example, methyl parathion (MHP) has been removed at rates of up to 80% and 90% when treated with cyanobacterium *Fischerella* sp. and *Microcystis novacekii*, respectively, through chemisorption between MHP and hydroxyl (-OH) groups on the cell surfaces [18, 19]. Chlorpyrifos has been effectively degraded through plasma treatment, which utilizes reactive oxygen species such as $\cdot\mathrm{O_2}$–, ·OH, and $1\mathrm{O_2}$ [20]. Additionally, it can be successfully removed from ripe fruits using a 1–2% citric acid solution [21].

Advanced oxidation techniques, such as electron beam radiation, have demonstrated their ability to degrade profenofos in both water and edible peas [22]. Additionally, biological approaches have been developed, such as the in vivo culture of *Acinetobacter* sp. 33F and *Comamonas* sp. 51F, which promoted the degradation of profenofos in soil through symbiotic interactions with *Vigna radiata* [23]. Adsorption-based methods have also been employed; for instance, activated carbon derived from corncob and rice husk successfully removed approximately 93% of dimethoate from water contaminated at 100 ppb concentration [24]. Gold nanoparticles have also been studied for their ability to absorb dimethoate, with research indicating effective pesticide removal through nanoparticle aggregation within 24 hours in drinking water [25].

Despite the proven effectiveness of these techniques, their feasibility for on-field applications remains a critical factor in determining the viability of each method. Plasma-based degradation of OP insecticides, although effective, requires sophisticated equipment and skilled personnel, making it impractical for widespread agricultural use. Similarly, microbial degradation relies heavily on environmental factors and the availability of essential nutrients to sustain microbial growth, which can be unpredictable in natural settings. Although activated carbon and gold nanoparticles have shown promising adsorption capabilities, the high cost of large-scale production presents a significant barrier to their implementation in developing regions and agricultural economies.

Therefore, ongoing research continues to focus on identifying more cost-effective, efficient, and environmentally friendly absorptive materials for the removal of agricultural chemicals. The development of novel materials with high adsorption capacities, reusability, and affordability is essential to addressing the organophosphate insecticides contamination. Future research should focus on integrating these materials into scalable and sustainable water treatment technologies with the economic and environmental constraints of affected regions.

III. MATERIALS AND METHODS

A. Materials

The commercial sodium bentonite (designated as bentonite) was purchased from Srichand United Dispensary Co., Ltd. (Bangkok, Thailand).

Berberine hydrochloride at pharmaceutical grade was provided by Shaanxi Yuantai Biological Technology Co., Ltd., China. Magnesium oxide (MgO) was supplied by Union Science Co. Ltd., Thailand.

Methyl parathion, chlorpyrifos, profenofos, and dimethoate (>97%, PESTANAL®) were purchased from Sigma and Riedel-de Haën (Germany) (Fig. 1). Stock solutions of insecticides were prepared in MeOH 100 mM and stored at 4°C prior to use. Working insecticide solutions were prepared at 5 ppm in distilled water (pH 7.2). All chemicals were of analytical grade.

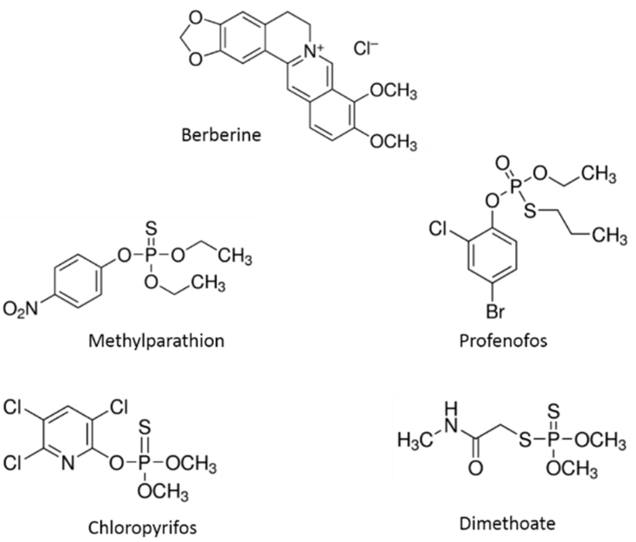

Fig. 1. Structure of berberine and tested organophosphate insecticides.

B. Fabrication of Absorptive Organobentonite/MgO Ceramic Composites Balls

The berberine-bentonite/MgO composite powder was prepared following a two-step process (adopted from [14]): (1) Na-Bentonite was first modified with pharmaceutical-grade plant alkaloid berberine. A total of 3 kg of Na-Bentonite was dispersed in 20 L of distilled water and mixed with 100 g of berberine (dissolved in 1 L of 95% ethanol). The mixture was continuously stirred at 500 rpm for 8 hours at room temperature to obtain berberine-modified bentonite, also referred to as organobentonite; (2) Magnesium oxide (MgO) was then added to the aqueous berberine-bentonite mixture at a concentration of 1.5% (w/w) (i.e., 45 g of MgO). The mixture was stirred at approximately 600 rpm for 5 hours. The resulting material was dried at 90°C for 120 hours and then ground into fine powder for the subsequent fabrication of organobentonite ceramic composite balls [26].

The organobentonite composite balls (Fig. 2) were fabricated using a granulation process typically employed for bentonite clay, incorporating appropriate additives such as kaolin, sinter, coal dust, corn starch, and starch. The granulation was carried out using a Sew-EuroDrive – Model R67AM56 Pan Granulator, following the method described by [27]. The firing process to produce the ceramic composite balls was carried out in collaboration with Manh Dan Ceramic Co. Ltd. (Gia Lam, Hanoi City, Vietnam). The firing was performed at 500°C for 6 hours, followed by incubation at 50°C for 12 hours and cooling under ambient air conditions.

The porous ceramic balls were expected to exhibit permeability of approximately 1.5×10^6 mD, as described in previous reports.

C. Fine Micronano Bubbles Water Generation

Fine Micronano Bubbles (MNBs) were generated using an air-water circulation system. A RGXM 3 high-pressure water pump (Speroni, Italy) was connected to an eco-bubble water generator MB0003SS (Strongco, Taiwan), which was immersed in 20 L of tap water in a glass tank (120 cm × 40 cm × 50 cm). The system was operated for 15 minutes at room temperature to generate MNBs. The characterization of MNBs was performed using a NanoSight NS300 instrument (Malvern Panalytical Ltd., United Kingdom).

The MNB generator was connected to the water volume in the tank and operated for 30 minutes to generate MNBs from air. The dissolved oxygen concentration was monitored over time using a Seven2Go™ DO meter (Mettler Toledo, United States).

D. Runoff Water Model Study for Organophosphate Insecticides Removal

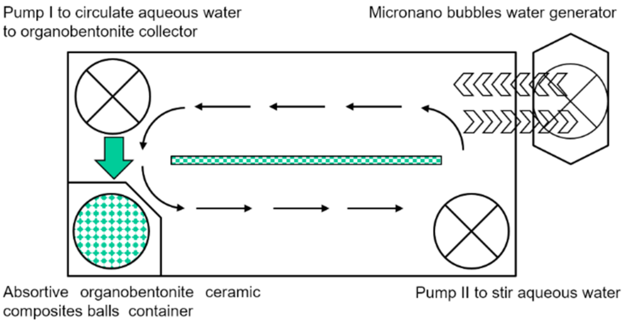

Fig. 2. Runoff model for the removal of tested OP insecticides in tap water.

The MNB water was spiked with organophosphate insecticides at a concentration of 5 ppm. The experimental tank was divided into two parallel channels by a partition wall (Fig. 3). Adjustable pump I was installed at one end to pump and circulate water through a plastic container filled with 4 kg of absorptive ceramic composite balls. This container was connected to an output faucet, as shown in Fig. 4D. Different flow rates of pump I, set at 40 L/min, were tested. Pump II was installed at the opposite end to regulate water flow in a runoff water system with a stream velocity of approximately 60 meters/min (Fig. 3).

The model was operated continuously, and water samples were collected at intervals of 6, 12, 18, and 24 hours to assess the persistence of the tested insecticides, the absorption capacity of the materials, and the overall efficiency of the removal system. The insecticide residues were analyzed using high-performance liquid chromatography (HPLC), as described in the following section.

E. Analysis of OP Insecticides by High Performance Liquid Chromatography

HPLC was used to determine the residual concentrations of organophosphate insecticides after treatment, following the method described by Anh et al. (2011). Aqueous samples (1 mL) were centrifuged at 20,627g for 15 minutes and filtered using a VeriPure Nylon Syringe Filter (0.2 µm pore size; Vertical®). The filtered samples were then transferred to 2 mL HPLC vials for analysis.

The HPLC analysis was conducted using an Agilent HPLC system (Series 1100; Agilent) equipped with a VertiSep™ AQS reversed-phase (C18) column (4.6 mm × 150 mm, 5 mm; Vertical®). A gradient elution method was applied, with acetonitrile concentrations varying from 20% to 80% over a 20-minute runtime (0–4 min: 20%; 4–12 min: 80%; 12–17 min: 20%). The flow rate was maintained at 1 mL min^{-1}. Insecticide residues were identified and quantified by comparison with authentic standards.

IV. RESULT AND DISCUSSION

A. Fabrication of Organobentonite Ceramic Composites Ball

The organobentonite ceramic composite balls are yellowish, porous, and approximately 2 cm in diameter, weighing around 7.2 g. The other physical properties of the composites are similar to those of other commercial porous ceramic balls used for water treatment, following the professional fabrication process of the manufacturer. Water can penetrate through the filter ball container efficiently at flow rates of 10, 20, and 40 L/h (Fig. 3D).

MgO was incorporated into the composites as an antibacterial and yeast-inhibiting reagent to counteract possible microbial contamination (Nguyen et al., 2018). Additionally, it helps prevent defluoridation (Thakre et al., 2010) and the precipitation of humic acid (Rasuli and Mahvi, 2016) when the ceramic balls are immersed in water.

The yellow appearance of the ceramic balls may be attributed to two factors: the berberine-modified bentonite, which is naturally yellow due to berberine, and the kilning process in the furnace used for ceramic ball fabrication [28].

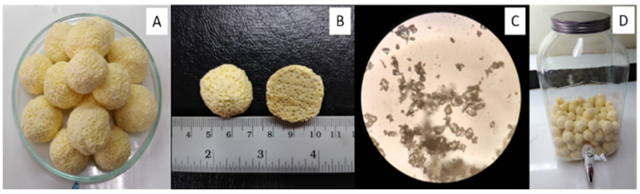

Fig. 3. The organobentonite ceramic composite balls. A: Final ceramic composite balls; B: Profile image of the balls; C: Ceramic composite crystal under a light microscope at 40× magnification; D: Container for absorptive balls.

The organo bentonite ceramic composites balls product is yellowish, porous and approx. 2 cm diameter and 7.2 g. The other physical properties of the composites is similar to other commercial porous ceramic balls for water treatment following professional fabrication of the manufacturer. The water can penetrate through the filter ball container fluently at 10, 20 and 40 L/h (Fig. 3D). MgO was incorporated into the composites as antibacterial and yeast reagent to against the possible contamination of microorganism (Nguyen *et al.* 2018) as well as to prevent the certain deflouridation (Thakre *et al.* 2010) and the precipitation of humic acid (Rasuli and Mahvi 2016) content during the ceramic balls are immersed in the water. The yellow appearance of ceramic ball might come from both factors of berberine modifying bentonite which is naturally yellow by berberine and the kilning in the furnace for making of the ceramic balls [28].

B. Morphological Characterization of MNBs

The MNBs have mean and mode sizes of 193.9 ± 13.7 nm and 107.7 ± 2.3 nm, respectively. The D10, D50, and D90 values were 98.2 ± 1.8 nm, 152.7 ± 6.0 nm, and 357.9 ± 59.7 nm, respectively. The density of MNBs was approximately 7 × 10^8/mL ± 1 × 10^7. The dissolved oxygen (DO) concentration in the water increased rapidly from approximately 7 mg/L to 26 mg/L within the first 9 minutes of MNB generation and reached 30 mg/L after 30 minutes.

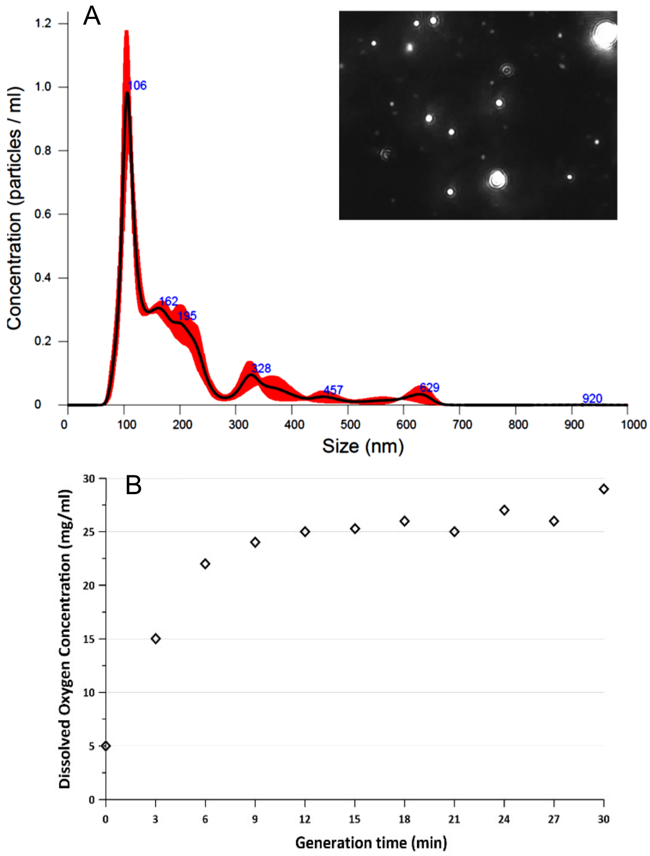

Fig. 4. Evaluation of MNBs water generation. A: Correlation between MNBs densities and their size; inset is MNBs still image from video streaming; B: Dissolved oxygen concentration determination in MNBs water.

C. Occurrence of OP Insecticides in the Water Runoff Model

Previous studies have reported that modified bentonite in mild powder form absorbs OP insecticides in tap water [14]. In this study, modified bentonite was fabricated into ceramic balls to facilitate field application. The concentration of each spiked OP insecticide in the water tank was analyzed over time at intervals of 6, 12, 18, and 24 h.

HPLC analysis revealed a linear decrease in all tested chemicals. The reduction percentages for chlorpyrifos were approximately 15%, 32%, 40%, and 42% at each respective time interval. Methyl parathion decreased by 10%, 25%, 30%, and 38%; profenofos by 18%, 35%, 40%, and 45%; and dimethoate by 8%, 20%, 30%, and 30%. These results indicate that our model functions effectively as an insecticide removal system. The residual concentrations of each insecticide over time are illustrated in Fig. 5.

Additionally, the modified bentonite in ceramic form retained its absorptive capacity for OP insecticides, and its absorptive structure remained unaffected by the heat treatment process. Studies have shown that treating native bentonite at 300°C and 650°C for 6 and 4 hours, respectively, can increase its internal surface area by enlarging its porous structure [29, 30]. Furthermore, bentonite modified with 0.5 M hydrochloric acid and heated at 500°C has been shown to effectively absorb herbicides such as atrazine, diuron, 2,4-dichlorophenoxyacetic acid, and paraquat.

The berberine-modified bentonite facilitates the intercalation of berberine molecules between bentonite layers, resulting in the expansion of the bentonite layers and the creation of additional hydrophilic sites on the surface, where exchangeable cations such as Na+ and Ca2+ are located. This modification enhances the ability of bentonite to absorb OP insecticides [14]. The structure of the modified bentonite was observed to remain unchanged after similar heat treatment conditions for SEM sample preparation [14, 26].

The incorporation of an MNB generator in the aqueous system contributed to the degradation of OP insecticides. This degradation is attributed to the mechanical shearing force of MNB water [31, 32]. Studies have shown that MNB diameters in the range of approximately 40–190 nm can remain stable for several days, while those between 250–350 nm persist for at least 24 hours [33, 34]. Additionally, the physical properties of MNBs, such as zeta potential and thermodynamic stability, remain largely unchanged at temperatures ranging from 20°C to 70°C [35, 36].

Within the experimental timeframe, the removal of tested OP insecticides was observed to occur within 24 hours.

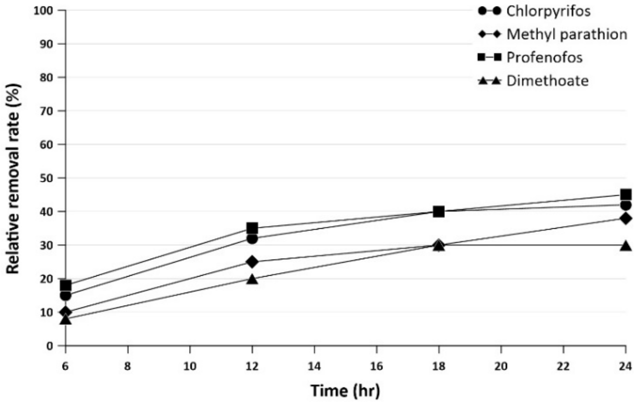

Fig. 5. The removal rates of tested OP insecticides by using modified bentonite ceramic balls and micronano buble water in the designed water runoff model

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V. CONCLUSION

The clean-up of OP insecticides in run-off water was carried out using berberine-modified bentonite-based ceramic balls as a chemically absorptive material in coordination with nano-bubble water, which acted as a "macromolecule cutting scissor." The elimination of tested

OP insecticides was recorded over the experimental time course. The fabrication of berberine-based ceramic balls did not alter the absorptivity of modified bentonite. This facilitated the use of bentonite in a solid form that can be applied to various aquatic terrains contaminated by agricultural chemicals. The porosity and surface characteristics of the ceramic balls allow for effective adsorption and interaction with pollutant molecules, improving the overall efficiency of water purification.

The micro-nano bubble water can be generated using various physical techniques, including cavitation, gas-liquid mixing, and electrolysis. The nano-bubbles enhance the degradation of organic pollutants by increasing the availability of reactive oxygen species, thereby improving the breakdown of OP insecticides. Accordingly, further applications of these materials should be explored to develop practical environmental green tools for the recovery of contaminated water zones. Next research will focus on optimizing the synthesis conditions of the ceramic balls, enhancing their stability, and investigating their long-term performance under real-world environmental conditions. Additionally, the combination of nano-bubble technology with other advanced oxidation processes could further enhance the removal efficiency of persistent pollutants in various water treatment.

Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Evaluation and analysis of the fabrication of related materials and the organophosphate insecticides degradation, writing, and preparation were performed by Kanchana Dumri and Dau Hung Anh. Model simulation was designed and applied by Dau Hung Anh. Kanchana Dumri and Chinanat Witthayaprapakorn conducted the conceptualization, supervision, resources management, validation, editing and writing manuscript. All authors approved the final version.

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