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Review Article

Ultrasonic and Electrocoagulation Technologies in Wastewater Treatment and Material Circulation

Nguyen Manh Khai[1,*], Nguyen Thi Mai[1,2], Tran Thi Minh Hang[1], Dang Van Thanh[3], Pham Tien Duc[1], Hoang Minh Trang[1]

[1]*VNU University of Science, 334 Nguyen Trai, Thanh Xuan Hanoi, Vietnam*
[2]*Thai Nguyen University of Agriculture and Forestry, Quyet Thang, Thai Nguyen, Vietnam*
[3]*TNU University of Medicine and Pharmacy, 284 Luong Ngoc Quyen, Thai Nguyen, Vietnam*

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Abstract: Due to the emergence of new and persistent pollutants in wastewater, various advanced oxidation processes (AOPs) such as ultrasonic technology and electrocoagulation have been developed and employed for pollutant degradation, removal, and recovery from wastewater. In this study, we investigated the principles, influencing factors, and practical applications of several methods combining ultrasound and electrocoagulation with catalysts and demonstrated the efficiency of each method. Additionally, we analyzed the challenges associated with these ultrasonic methods and electrocoagulation in removing antibiotics and recovering pollutants from aqueous solutions and suggested solutions to these problems. Materials recovered after treatment can be utilized to improve soil quality.

Keywords: Ultrasonication, electrocoagulation, antibiotics, ammonia, phosphate.

1. Introduction

In recent years, a series of emerging pollutants such as pharmaceuticals, perfluorinated compounds, personal care products, manufactured nanomaterials, and endocrine disruptors, etc., [1] have attracted significant attention. Although these substances typically appear in very low concentrations in water, many studies have suggested their negative impacts on aquatic creatures [2] which subsequently affected human health. Additionally, almost all of these contaminants are difficult to degrade using conventional

* Corresponding author.
E-mail address: nguyenmanhkhai@hus.edu.vn

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methods. Therefore, advanced oxidation processes like ultrasonication, electrocoagulation, electrochemistry, photocatalyst, Fenton, etc., have been applied for decomposing these compounds due to their high removal efficiency and short treatment time. Among these, ultrasonication and electrocoagulation are two of the most popular methods because of their advantages.

Electrocoagulation, a technique combining electrochemistry, coagulation and flotation, is usually easy to operate, low capital costs, high removal effectiveness, sludge limitation, automation ability, handle multiple contaminants, no addition of chemicals and short/moderate operating time [3, 4] whereas ultrasonic treatment is an more efficient and time-saving treatment than conventional treatment [5], simple and low investment costs [4]. However, ultrasonication is often not highly effective without support of other technologies or catalysts, while electrocoagulation may exhibit limitations such as passivation electrodes, high energy consumption and dissolution of electrodes. Besides that, electrolytes need to be added due to poor electrical conductivity of water. Additionally, electrocoagulation is not effective with contaminants which is not able to form precipitates or sludges.

To address these problems, the combination of ultrasonication and electrocoagulation with other techniques or with catalysts was suggested. Several studies have combined ultrasonication with electrocoagulation [4, 6-9], photocatalyst [10], Fenton [11], catalyst [5] or electrocoagulation with electroflotation, flotation [12], ozonation, UV light [13], Fenton [14] etc., for enhancing the removal efficiency. Additionally, several pollutants collected after treatment were used as materials for enhancing the soil quality. Phosphate collected from wastewater was used as alternative source of agricultural phosphorous, one necessary element for agricultural production [15]. Struvite ($NH_4MgPO_4.6H_2O$) which was used as one kind of slow-release fertilizer, was used to adsorb N and P from pig farming wastewater supplying N (6%), P_2O_5 (28.9%) and Mg (10%) for many crops [16, 17].

In the research, the principle, effecting elements and actual cases of ultrasound-based and electrocoagulation-based technologies are analyzed. Simultaneously the issues of these methods in the degradation and removal of antibiotics (ciprofloxacin, sulfamethoxazole, levofloxacin, enrofloxacin, amoxicillin, tetracycline,…) and the recovery of compounds (ammonia, phosphate,…) from wastewaters also examined, and handling methods are indicated.

2. Ultrasonication Technology

Ultrasound-based operates on the principle of acoustic cavitation in which the sound waves generate compression and rarefaction cycles while transmitting through liquid medium. Small vacuum bubbles are produced which then fall down within a few microsecond after reaching a threshold size during alternating high-pressure (compression) and low-pressure (rarefaction) cycles [18]. The bubbles continue the growth until attain a volume at which they can no longer absorb energy, they collapse violently during compression cycle generating high pressure (500 bar) and high temperature (5000 ^oC) (Fig. 1).

At this condition, the H_2O and O_2 molecules are excited forming free radicals $^{\bullet}OH$ and $^{\bullet}O_2^-$ (Eq. 1-2) [19].

$$H_2O + \text{ultrasound} \rightarrow {}^{\bullet}OH + {}^{\bullet}H \quad \text{(Eq. 1)}$$

$$O_2 + \text{ultrasound} \rightarrow {}^{\bullet}O_2^- \quad \text{(Eq. 2)}$$

The produced hydroxyl and peroxide radicals are highly reactive due to their high oxidation-reduction potential thus they improve oxidation reactions more than traditional oxidants. Ultrasound is simple and effective technology with commonly low investment costs, however it is often not very high effective as single water treatment, thus it is usually combined with catalysts, Fenton, photocatalyst, ozone, electrochemical processes, etc [20].

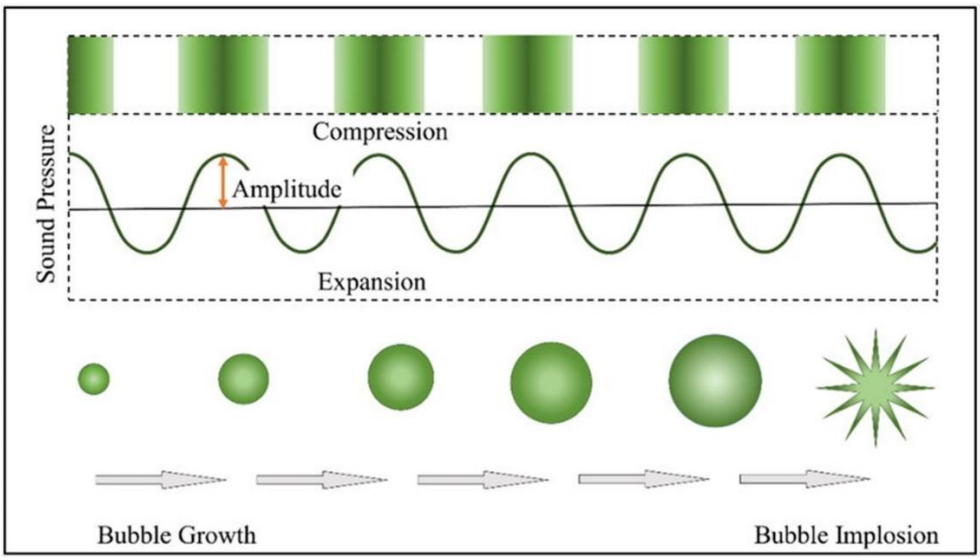

Figure 1. Principle of ultrasound cavitation [18-21].

2.1. Sonocatalysis

Sonocatalysis is the combination between sonochemistry and catalysis for the accomplishment a series of degradation reactions with more convenient conditions such as shorter reaction time, higher degradation effectiveness, lesser consumed energy, effectively oxidize organic contaminants [22, 23], beneficial handling and low cost and environmental friendliness [30] than traditional methods. It does not require pre-treatment of the effluent and the penetration capacity of ultrasound waves is strong during degradation process of contaminants by catalyst [24, 25].

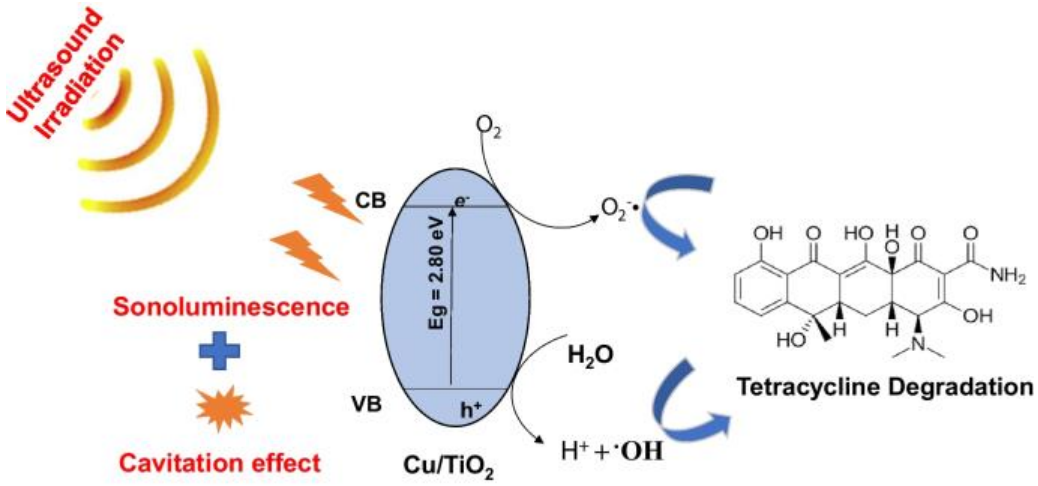

Figure 2. The proposed degradation mechanism of tetracycline by sonocatalysis using Cu/TiO_2 as catalyst [26].

The main steps of sonocatalysis is diffusion and sorption of major components on a catalyst surface followed by a series of oxidation reactions on active sites [27]. It is obvious that high pressure and high temperature generating due to ultrasound waves excited O_2 and H_2O

molecules dissolved in aqueous solution forming $^{\bullet}O_2^-$ and $^{\bullet}OH$ radicals. Spontaneously, the light emitted due to high temperature also excited the electrons (e^-) in valance band (VB) of catalyst [20] which then jumped to covalent band (CB) generating the holes (h^+) in VB. These holes excited OH^-/H_2O giving $^{\bullet}OH$ radicals whereas electrons accelerate O_2 indicating the $^{\bullet}O_2^-$ radicals [26]. $^{\bullet}O_2^-$ and $^{\bullet}OH$ radicals then initiated the degradation reactions for organic contaminants (Fig. 2). Thus, the process is convenient to treat wastewater containing a series of organic contaminants amongst others.

The generation of active radicals in sonocatalysis is exhibited using the equations below:

$$\text{Ultrasound} + \text{catalyst} \rightarrow e^- + h^+ \quad \text{(Eq. 3)}$$

$$h^+ + H_2O \rightarrow {}^{\bullet}OH + {}^{\bullet}H \quad \text{(Eq. 4)}$$

$$e^- + O_2 \rightarrow {}^{\bullet}O_2^- \quad \text{(Eq. 5)}$$

$$^{\bullet}OH + {}^{\bullet}OH \rightarrow H_2O_2 \quad \text{(Eq. 6)}$$

$$H_2O_2 \rightarrow 2{}^{\bullet}OOH + {}^{\bullet}H \quad \text{(Eq. 7)}$$

$$^{\bullet}OOH + \text{pollutants} \rightarrow \rightarrow\rightarrow CO_2 + H_2O \quad \text{(Eq. 8)}$$

$$^{\bullet}OH + \text{pollutants} \rightarrow \rightarrow\rightarrow CO_2 + H_2O \quad \text{(Eq. 9)}$$

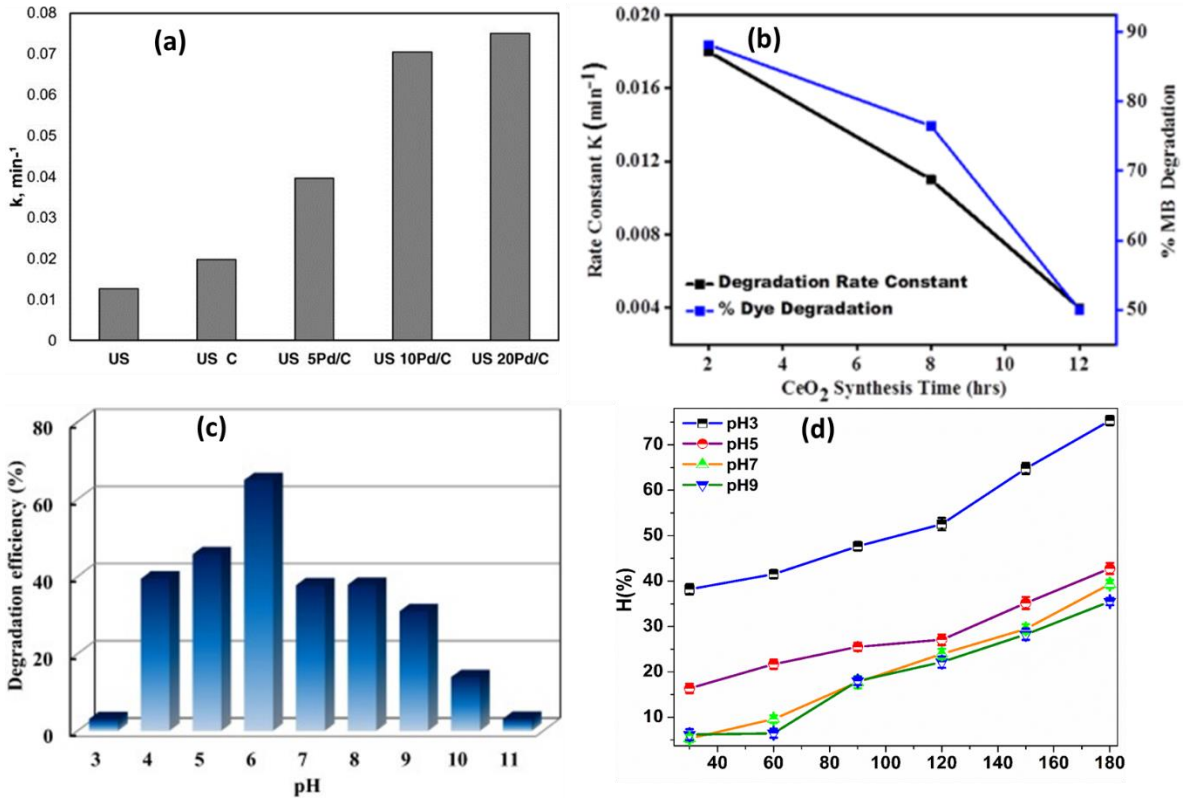

Figure 3. Degradation rate constant of butylparaben using Pd/C (a) [27], degradation rate constant and efficiency of methylene blue using CeO_2 spindles (b) [28] and degradation efficiency of ciprofloxacin using TiO_2 - coated montmorillonite (c) [29], degradation efficiency of SMX using red mud/biochar [31]

Many catalysts were used to degrade organic compounds in combination with sonication. Bampos and Frontistis (2019) was used Pd/C nanoparticles for degradation of butylparaben in aqueous solution with the support of ultrasound [28]. The results illustrated that the removal rate of butylparaben increased considerably with the dose of 25 mg/L Pd/C. Besides, the kinetic constant of decomposition increased from 0.0126 to 0.071 min^{-1} and the process was being

favored under alkaline solution (Fig. 3a). Besides, methylene blue was degraded using CeO_2 spindles as a catalyst [30] with the aid of ultrasound. The degradation effectiveness achieved 90% after 2 h of treatment, and the rate constant was 0.018 min^{-1} (Fig. 3b). It reveals that CeO_2 can be used as an excellent catalyst for degradation of contaminants. Additionally, antibiotics, one kind of emerging pollutants which was studied and removed out of wastewater and ciprofloxacin, an antibiotic belonging fluoroquinolones group, was decomposed using TiO_2-coated montmorillonite combined with ultrasound. At pH 6, 0.2 g L^{-1} of catalyst, 10 mg L^{-1} of CIP and ultrasonic power of 650 W L^{-1}, degradation effectiveness was 65.01% (Fig. 3c). The stability of catalyst still remained after 4 recycle times [30]. Thus, the combination of sonication and catalyst improves the removal efficiency, enhances degradation rate and lowers the reaction time. N T Mai et al., (2024) synthesized nanomaterial using red mud and rice husk char as starting materials which then was applied for degradation of sulfamethoxazole in aqueous solution [31]. It can be seen in Fig. 3d, the degradation efficiency decreased with increasing of pH and the highest efficiency was 75% at pH 3 and contact time of 180 min. It may due to the presence of ferrous oxide in materials with high content thus the nanomaterial can act as Fenton agent [32]. At low pH, Fe^{2+}/Fe^{3+} can be easy to attach H_2O_2 to form $^{\bullet}OH$ and $^{\bullet}OOH$ radicals which initated the degradation reaction of SMX so the efficiency would increase sigfinicantly.

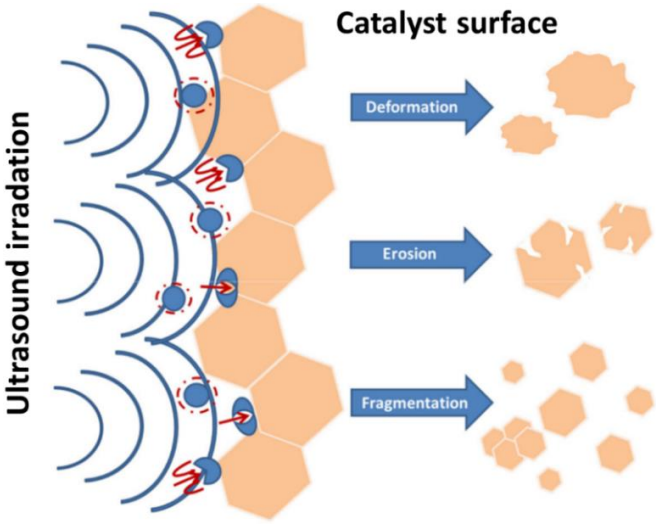

Figure 4. Effect of ultrasound irradiation on a catalyst surface [23].

However, in the sonocatalysis system using solid catalysts, the increase of oxidation reaction rate is mainly caused by physical phenomenon which enhance the mass transfer from disordered mixture and acoustic stream producing cavitation erosion at catalyst-solution interfaces being responsible for deformation of catalyst surfaces (Fig. 4). Therefore, the removal efficiency of pollutants decreases significantly after several recycling times. This point was proved in our research as being shown in Fig. 5.

In this research, red mud, a waste of aluminum production industry from Bauxite ore using Bayer technique, is recovered and recycled as raw material for synthesis of catalyst. Wet red mud was used as electrolyte to exfoliate graphene from graphite electrodes which was collected from released battery core obtaining red mud/graphene (RMG) catalyst. After that, RMG was used as catalyst for removal/degradation of CIP antibiotic in aqueous solution. The result in Fig. 5 indicated

the recycling of RMG as catalyst for eliminating CIP with the support of ultrasound. It is obvious, the efficiency increased with rising of sonication time and reached the maximum values after 180 min. However, the figures decreased slowly from 80.46% in the 1st cycle to 78.31% in the 2nd cycle, 72.25% in the 3rd cycle which then declined dramatically to 62.66% in 4th cycle and remained 50.22% in 5th cycle. The decrease can be explained that: i) The deformation of catalyst surface by cavitation erosion occurring during sonication process; ii) The filling of the pores on the catalyst surface by pollutants.

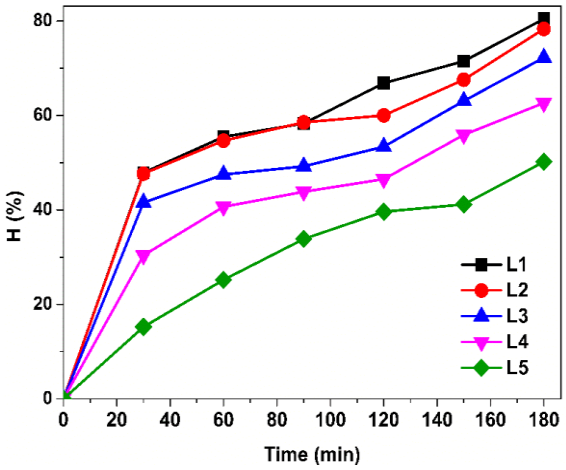

Figure 5. Recycle of red mud/graphene (RMG) as catalyst for eliminating of CIP with support of sonication.

2.2. Sono-electrocoagulation

To solve the cavitation erosion phenomenon of sonocatalysis, sonoelectrocoagulation (SEC), a hybrid process which is combined between sonication and electrocoagulation, is used. While electrocoagulation (EC) process, an inexpensive method for dealing with several wastewater and industrial effluents, can create positive species which causes for neutralizing the contaminant surface charges and maintains suspended, emulsified or dissolved pollutants formed attracting the opposite charge species and generate flocculants. However, a passive film can form on the surface of electrode (usually is Al or Fe) [33] during process, reasoning for increasing the operation time, energy consumption, and decreasing the removal efficiency. Besides that, this passive film also influences the generation rate of bubbles as well as coagulant. To deal with this problem, ultrasound was coupled with EC to: i) Break down sediments covered on the electrode surface; ii) Enhance the activation of electrode surface; iii) Decrease the reaction time by declining the diffusional resistance thickness at electrode; and iv) Produced high amounts of free radicals which initiated the degradation reactions of pollutants causing rapid process, high effectiveness, and cost-effective [34, 35].

According to various research, the principles of sonoelectrocoagulation, including three main stages, were illustrated in Fig. 6. i) When electricity is provided, the electrolytic oxidation reactions occur at anode leading to dissociation of metal ions producing the M^{n+} (coagulants) and e^-, at the same time, H_2O molecules are also electrolyzed to release O_2 and e^-. After that, e^- are transferred to cathode for reacting with H_2O molecules to form H_2 gas and OH^-; ii) M^{n+} ions destabilize contaminants, creates suspension, and breaks emulsions. Spontaneously, the electrochemical reduction of metallic ions can be occurred directly at cathode forming a thin film on surface of cathode whereas M^{n+} combines with OH^- making metal hydroxides ($M(OH)^n$) before depositing on the bottom; and iii) Destabilize phases (M^{n+}, pollutants (P) and OH^-) are aggregated producing flocculants before floating on the surface [36, 37]. Additionally, when ultrasound waves are applied, the high pressure and high temperature are generated which excites O_2 and H_2O molecules forming the $^{\bullet}OH$ and $^{\bullet}O_2^-$ radicals. These radicals initiate the degradation reaction chain of pollutants, thus the efficiency of wastewater treatment by sono-electrocoagulation process improves significantly. US waves can also preclude passivation of cathode by cavitation phenomenon.

The main reactions occurring during sono-electrocoagulation process were shown below:

$$M^0(s) - ne \rightarrow M^{n+}(aq) \quad \text{(Eq. 10)}$$

$2H_2O\ (l) + 2e \rightarrow H_2 + 2OH^-$ (cathodic reaction) (Eq. 11)

$2H_2O\ (l) \rightarrow 4H^+\ (aq) + O_2 + 4e$ (anodic reaction) Eq. 12)

$M^{n+} + ne \rightarrow nM^0$ (cathodic reaction) (Eq. 13)

$M^{n+} + nOH^- \rightarrow M(OH)_n$ Eq. 14)

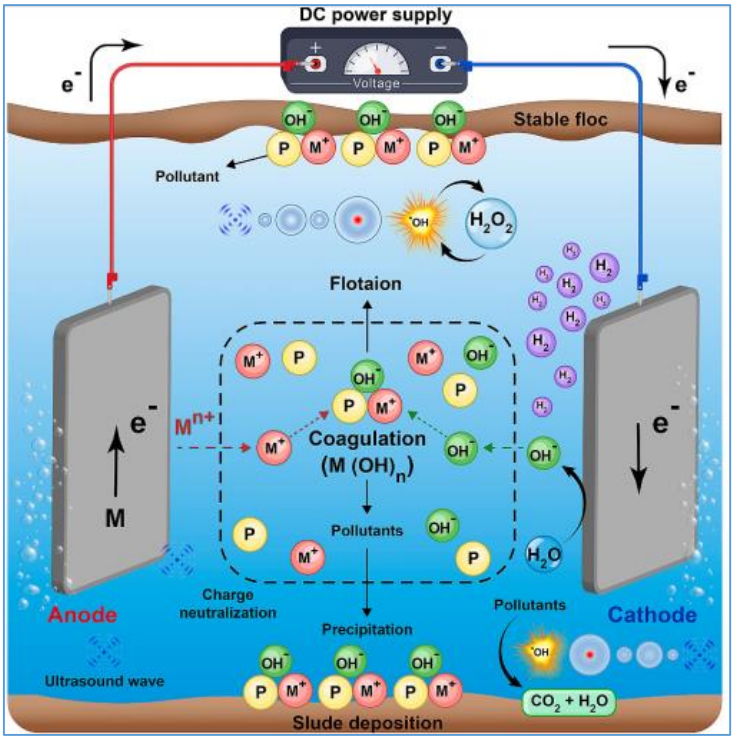

Figure 6. The scheme of the pollutants degradation mechanism in the ultrasound/electrocoagulation system [36].

Series of research concerning the SEC have been conducted. Sadeghi et al., used Ti/PbO_2 as the anode and Al as the cathode electrodes for removal of arsenic in the basic environment [38]. The results (Fig. 7a) demonstrate that the removal efficiencies of arsenic by sono-electrocoagulation were higher than that of electrocoagulation at all investigated concentrations. Thus, a couple of ultrasonic and electrocoagulation processes are a potential method for arsenic removal. Besides, Arka et al., (2022) investigated the effects of operation parameters of SEC process on removal efficiency of chemical oxygen demand (COD) and colors. The results proved that 97.5% of COD, 100% of colors were removed out of aqueous solution (Fig. 7b) at current density of 1 A dm^{-2}, pH of 5, electrolyte concentration of 6 g L^{-1}, an inter-electrode distance of 1 cm, the minimum power consumption of 0.55 kWh m^{-3} and reaction time of 45 min [39]. Moreover, Asaithambi et al., (2017) applied SEC for treatment of landfill leachate wastewater based on the evaluation of removal efficiency of color and COD along with consumption of power. The experimental results (Fig. 7c) indicated the removal effectiveness of color (100%) and COD (94%) by SEC at low power consumption (4.5 kWh m^{-3}) was higher significantly than individual US process and slightly higher than the figure for EC alone. At optimal conditions: electrolyte concentration of 0.76 g L^{-1}, current density of 2.75 A dm^{-2}, initial COD concentration of 3919.50 mg L^{-1}, ultrasound power of 100 W and experimental time of 36.05 min, the maximum COD removal effectiveness

reached 71.05% with minimum power consumption of 2.33 kWh m^{-3} [40]. Meanwhile, our research applied SEC for recover of phosphate from aqueous solution which then applied for regeneration of soil quality. The effect of pH was investigated in our research and indicated in Fig. 7d. It is obvious that the removal efficiency of PO_4^{3-} (using ferrous electrodes) decreased when pH rose from 5 (73.73%) to 7 (54.74%) which then increased significantly to 79.50% at pH 9 before declining to 53.59% at pH 10. Ferrous bar, which exists in different form at various values of pH (Eq. 15-20) [41, 42], is used as an electrode of sonoelectrocoagulation system. At pH≤6, ferrous exists in $Fe(OH)_2^+$ form which contains opposite charge with PO_4^{3-} thus it may be good coagulants for PO_4^{3-} causing high removal effectiveness whereas the form $Fe(OH)_3$ of ferrous at 6≤pH≤8 is poor coagulants. At 8<pH≤10, the $Fe_3(OH)_4^{5+}$ ion appears becoming greatly beneficial coagulant for PO_4^{3-} so the efficiency increased considerably to 79.56%. However, the number declined strongly to 53.59% when pH>10 due to the existence of $Fe(OH)_4^-$ form containing opposite charge with pollutants [43].

$$Fe + 2H^+ \rightarrow Fe^{3+} + H_2 \, (pH \leq 3) \quad \text{(Eq. 15)}$$

$$Fe^{3+} + 2H_2O \rightarrow Fe(OH)_2^+ + 2H^+ \, (3 < pH \leq 6) \quad \text{(Eq. 16)}$$

$$Fe(OH)_2^+ + OH^- \rightarrow Fe(OH)_3 \quad (6 \leq pH \leq 8) \quad \text{(Eq. 17)}$$

$$3Fe(OH)_2^+ \rightarrow Fe_3(OH)_4^{5+} + 2OH^- \, (8 < pH \leq 10) \quad \text{(Eq. 19)}$$

$$Fe_3(OH)_4^{5+} + 8OH^- \rightarrow 3Fe(OH)_4^- \quad (pH>10) \quad \text{(Eq. 20)}$$

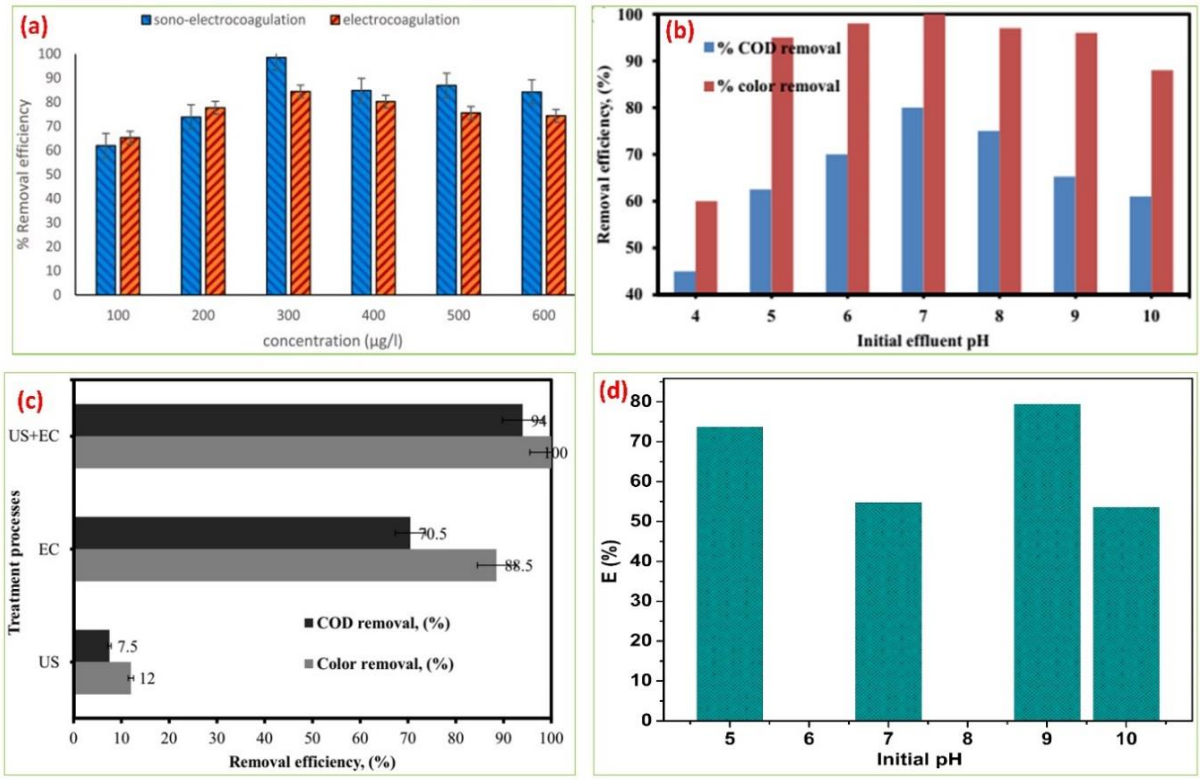

Figure 7. Effect of concentration on removal efficiencies of arsenic (a) [37], effect of initial pH (b) [38] and treatment processes on removal efficiency of COD and color (c) [39] , effect of pH on removal efficiency of PO_4^{3-} (d) (our research) by sonoelectrocoagulation.

3. Recover of N and P from Wastewater for Supplement Nutrients for Crops

Nitrogen and phosphorus are non-renewable ore resources and become depleted due to higher consumption in agriculture. Moreover, the N and P can be removed out from wastewater treatment plants, municipal sewage [44] as well as from unabsorbed fertilizers causing water eutrophication [15]. Therefore, finding the way to recover N and P to supply for crops is one beneficial solution for reducing environmental pollution as well as improving the quality of soil. A series of methods for collecting N and P were suggested. N-P fertilizer was synthesized using N-rich slurry from pig farms and P-rich incineration fly ash from chicken farms (Fig. 8). MgO was supplied as a magnesium source for struvite precipitation (following Eq. 21-24 (FA-MAP)) [43], one kind of slow-release fertilizer, which contain 6% N, 28.9% P_2O_5 and 10% Mg. This fertilizer is suitable for many crops thus struvite was used as material for production of mixed fertilizer [45].

$$MgO + H_2O \rightarrow Mg^{2+} + OH^- \quad \text{(Eq. 21)}$$

$$Mg^{2+} + NH_4^+ + HPO_4^{2-} + 6H_2O \rightarrow MgNH_4PO_4 \cdot 6H_2O\downarrow + H^+ \quad \text{(Eq. 22)}$$

$$Mg^{2+} + NH_4^+ + H_2PO_4^- + 6H_2O \rightarrow MgNH_4PO_4 \cdot 6H_2O\downarrow + 2H^+ \quad \text{(Eq. 22)}$$

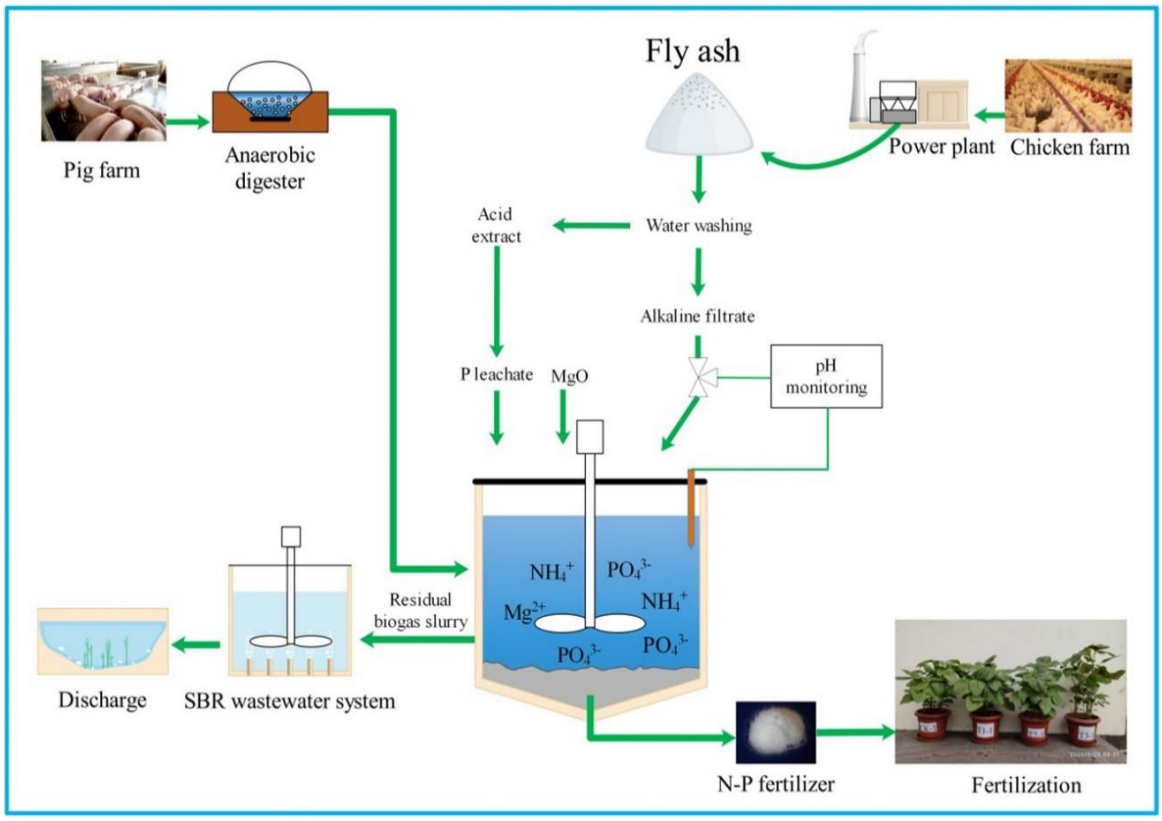

Figure 8. Recover N-P fertilizer from biogas slurry [43].

This product was mixed with soil before growing Amaranth seedlings. The results indicated in Fig. 9 proved that the Amaranth seedlings fertilized by FA-MAP had the highest height (about 23 cm) and chlorophyll (37.5 SPAD index unit) leading to the highest dry weight (8.2 g), demonstrating the effective solution for material circulation.

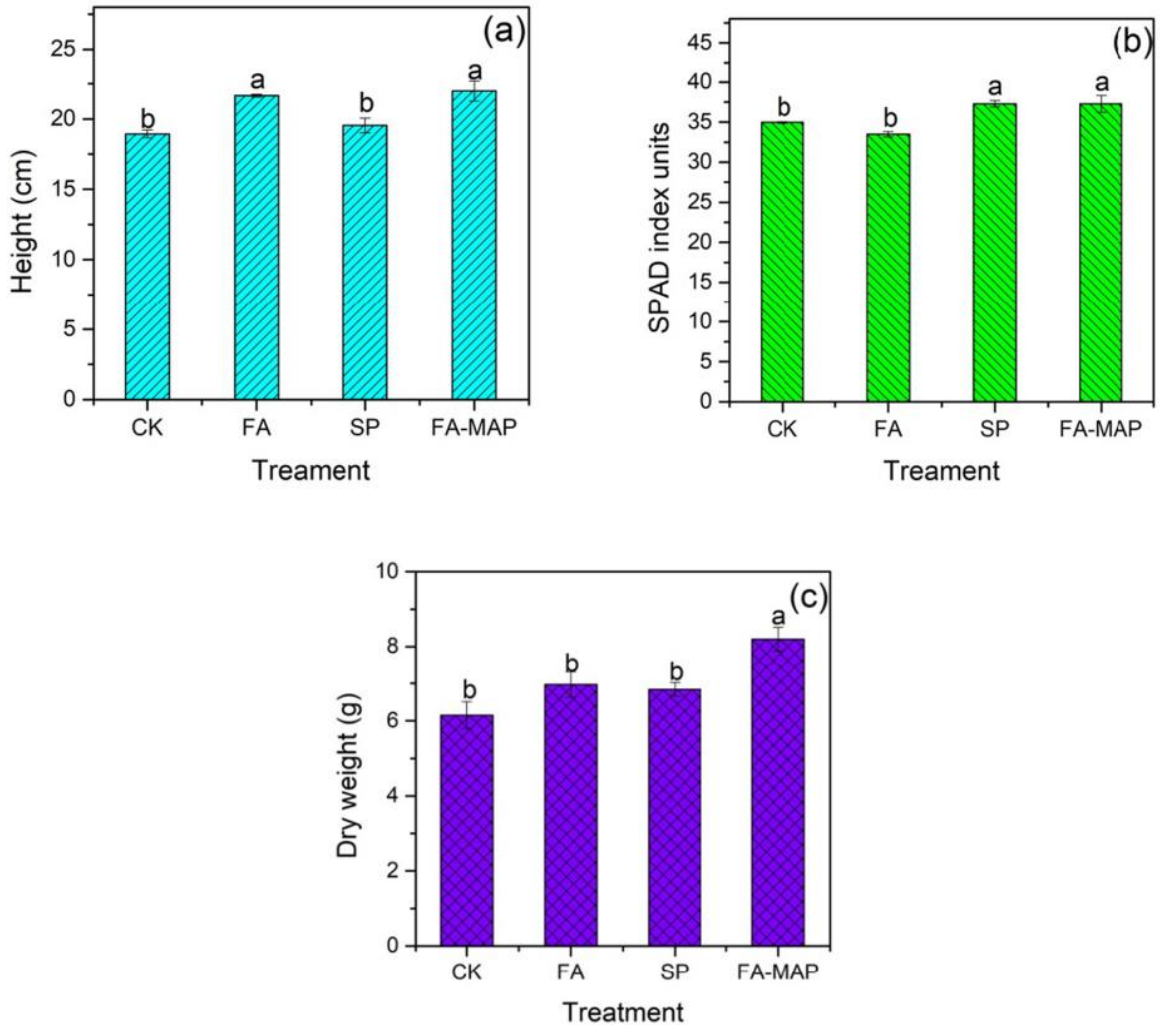

Figure 9. Amaranth biomass (a: Height; b: SPAD index units; c: Dry weight of aboveground part) Note: CK: no phosphate fertilizer; FA: phosphorus-rich fly ash; SP: superphosphate; FA-MAP: N-P fertilizers [43].

Luo et al., (2022) used constant voltage electrocoagulation with magnesium anode to crystallise struvite from chicken manure slurry which digested anaerobically (Fig. 10). Following the 2nd order kinetic model, maximum rate constant of phosphorous recovery is about 2.13 h^{-1}. The recovery rate of 5.24 mgP/h/cm^2 reached at voltage of 2 V, temperature of 25 ^oC, digestion time of 90 min and 4.60 mgP/h/cm^2 in anaerobic treatment system of chicken manure. It indicated that the purity of recovered struvite was 98.9% at initial pH of 7.9 [46]. Thus, the magnesium electrode was able to rapidly combin with NH_4^+ and PO_4^{3-} to produce high quality struvite.

Bhoi et al., (2023) developed an electrocoagulation system for the precipitation of phosphorus from anaerobic effluent using iron cathode and magnesium anode (Fig. 11). The results showed that 98.05% TP was precipitated at pH of 6.75, recovery time of 11.06 min, current density of 300 A m^{-2}, electrode distance of 1.5 cm and power consumption of 1.28 kWh m^{-3} [47]. The rate constant of TP precipitation following 1st order kinetic was 0.185 m min^{-1}.

Then, precipitated phosphorus was combusted at 900 °C followed by leaching using sulfuric acid. Around 91% of phosphorus was recovered at a liquid: solid ratio (v/w) of 100 mL/g. Besides, Mg^{2+} ions collecting from anode also combined with PO_4^{3-} and NH_4^+ in electrolyte solution forming struvite following Eq. 22. However, the effect of electrode passivation and polarity reversal on operation of electrocoagulation system have not investigated. Therefore, the continued studies should carried out to give optimal conditions for recovery of N and P from anaerobic effluent.

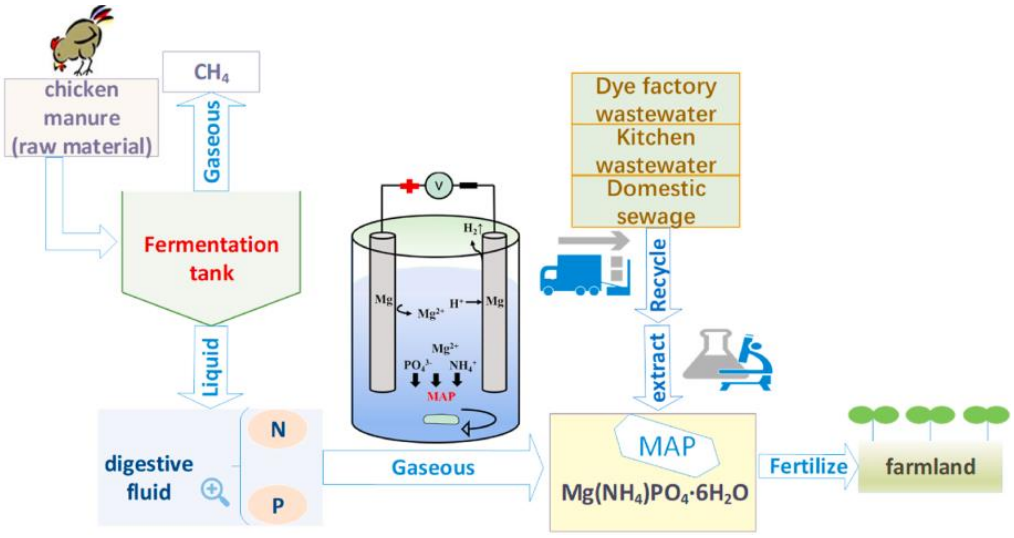

Figure 10. Production of struvite by magnesium anode constant voltage electrolytic crystallization from anaerobically digested chicken manure slurry [46].

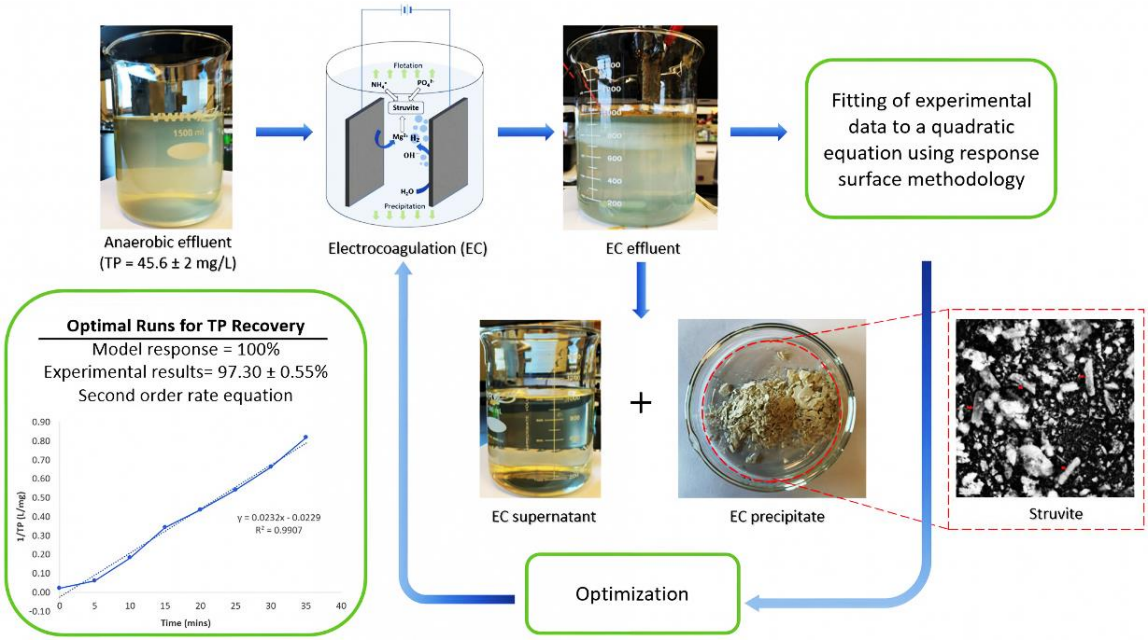

Figure 11. Electrocoagulation system for the precipitation of phosphorus from anaerobic effluent [47].

4. Conclusion

Both sonocatalysis and sonoelectrocoagulation can increase considerably the removal efficiency of contaminants (color, COD, PO_4^{3-}, etc.,) in aqueous solution rather than electrocoagulation or ultrasound alone or conventional methods. Whereas sonoelectrocoagulation is also an effective method for recovery of N, P from wastes which then was used as components of struvite fertilizer. Thus, these processes illustrated satisfactory results in removal a wide range of pollutants from solids, liquids, turbidity, COD to TOC. Besides high effectiveness, the promising processes are adequate operation and maintenance cost, short treatment time, high settleability and low sludge generation without supplementation of any chemicals. The research proves that sonocatalysis and sonoelectrocoagulation have high potential in removal of pollutants, recovery and circulation of material for crops.

Acknowledgements

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