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Application Of Micro-Nano Bubbling Aeration Method To Enhance Aerobic Process Efficiency In Winery Wastewater Treatment

A Project White Paper

July 2021

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Date:

March 2021

Research Team

Lubo Liu

Dr. Lubo Liu is a registered professional engineer in Environmental Engineering and a full professor at the Department of Civil and Geomatics Engineering in the Lyles College of Engineering of California State University, Fresno. He got his B.S. and M.S. degrees from Tsinghua University of China and a Ph.D. from the University of South Carolina in Civil Engineering. His research mainly focuses on water resources and environmental engineering. His specific research experience has been in the areas of reactive transport and the fate of contaminants in surface water and groundwater, environmental process modeling, groundwater bioremediation, subsurface characterization, and soil remediation. He is particularly interested in integrating laboratory and field-scale observations with modeling to explore the relevant processes and parameters.

Dr. Liu has widely published his research findings in journals and conferences in Environmental Engineering and Water Resources Engineering. He is currently devoting most of his research effort to two aspects: 1. A NSF-funded research project on how to improve the capability of water retention and nutrient in agricultural soil, and 2. A project on understanding near-shore processes in the Great Lakes and the transport of pathogens in the environment.

Zhi Liang

Dr. Zhi Liang is an Associate Professor of Mechanical Engineering in the Department of Mechanical Engineering of California State University, Fresno. He recieved his bachelor and master degree on Materials Science and Engineering from Shanghai Jiaotong University in Shanghai, China in 2001 and 2004. He earned his doctorate degree om Mechanical Engineering from Missouri University of Science and Technology in Rolla, Missouri in 2010. Dr. Liang's research interests include micro/nanoscale thermodynamics and heat transfer, nanofluidic energy conversion and storage devices, structure-property relationship for interfaces, snd computational modeling.

Yuanyuan Xie

Dr. Yuanyuan Xie is an Assistant Professor working in the Department of Mechanical Engineering of California State University, Fresno. He earned his doctorate degree from the University of South Carolina, Columbia in the year of 2013. Prior to joining Fresno State, he served as a postdoctoral researcher at Chemical Science and Engineering Division of Argonne National Laboratory. His research spans from electrochemical system, water treatment, micro/nano materials, energy storage and conversion, to multi-physicochemical modeling. Dr. Xie has produced more than 30 journal papers and given over 10 presentations in national / international conferences and technical meetings.

At Fresno State, Dr. Xie served as a faculty fellow for the California Water Institute (CWI) in the academic year 2020-2021. He is also an active member of the Electrochemical Society, the Material Research Society and the American Society of Mechanical Engineering.

Qun Sun

Dr. Qun Sun is an Assistant Professor of Enology in the Department of Viticulture & Enology of California State University, Fresno. She recieve her Food Science bachelor and master degree from Southwest Agricultural University, China in 2002 and 2005. She earned her doctorate degree of Wine Chemistry from Cornell University in the year 2011. She has had over 9 research projects since 2014. Dr. Sun has given multiple presentations as well as written various publications.

Problem Statement

This research utilizes micro-nano bubbling (MNB) method to enhance oxygenation capability for aeration to further increase the COD/BOD removal efficiency for wine wastewater treatment. A completely stirred tank reactor (CSTR) in a pilot scale will be designed and built in the labs of the LCOE and JCAST to simulate the aerobic process in winery wastewater treatment. Batch and CSTR experiments combined with mathematical modeling will be conducted to **1)** investigate the winery wastewater properties in central California, **2)** quantitatively differentiate the dissolved oxygen (DO) level and retention time in the aeration tank compared with the traditional aeration methods, **3)** investigate the impact of the MNB aeration rate on COD/BOD removal efficiency, **4)** determine the aeration rate coefficient, simulate and predict the electrochemical processes in the aeration tank with the MNB aeration by using an electrochemical model, **5)** explore and rank the impacting factors for the COD/BOD removal efficiency (sensitivity analysis), and **6)** optimize the operating factors in consideration of maximizing aeration efficiency and minimizing the cost.

Technical Background

Wine production is one of the most important agricultural activities and leading sectors in the food process industry worldwide, especially in California (Bolzonella, Papa et al. 2019, Maicas and Mateo 2020) . Although traditionally considered an environment-friendly process, wine production generates a large amount of wastewater and organic wastes due to the considerable consumption of water, fertilizers, pesticides, and organic amendments (Lofrano and Meric 2016) . In addition, cleaning the equipment and machinery may produce a significant amount of wastewater. The wastewater needs to be appropriately treated before it is discharged into receiving waters or reused (Novo 2021) .

In California, the wine industry contributes about $57.6 billion in revenue to the state's economy every year. However, process water from winemaking can degrade the water receiving body's quality because of its nitrogen content, salinity, and organic contaminants. The industry has faced unprecedented challenges since 2020 with substantial losses from wildfires and the pandemic. California has recently promulgated more stringent regulations on winery wastewater treatment and discharge. The winery industry is being forced to change the traditional land-based wastewater treatment and disposal system due to environmental concerns associated with organic, salt, nitrogen, and odors. It is foreseeable that this type of change will add costs to the winery wastewater treatment process (Chrobak 2002) . <u>Therefore, it is necessary and urgent to develop a treatment technology for winery wastewater that will both lower the economic cost of treatment and improve the quality of the treatment.</u>

The winery wastewater typically contains a high concentration of biodegradable organic compounds, phenolic compounds, sugars, ester, glycerol, organic acids, nutrients, etc. These compounds are mainly generated from washing operations during grape harvesting, pressing, and fermentation (Basile 2015) . The concentration of the organic matter is measured by total organic compound (TOC), dissolved organic carbon (DOC), biochemical oxygen demand (BOD), and chemical oxygen demand (COD). The concentration of COD or BOD in winery wastewater is usually much higher than that in municipal wastewater. They will be the main parameters used to quantify the level of organic compounds in the wastewater for water quality analysis in this research.

Among several well-developed methods to remove COD/BOD reported in the literature, aerobic processes are widely applied in both pretreatment and secondary treatment (such as activated sludge treatment) for their high removal efficiency and ease of use. In aerobic processes, aeration delivers oxygen for biochemical reactions and plays a significant role in controlling the COD/BOD removal efficiency. A good aeration unit may lead to a very high COD/BOD removal efficiency (Bolzonella, Papa et al. 2019) . As a key component of the treatment system, an aeration unit significantly affects the operation cost of the treatment. The literature has reported many studies on aeration processes in wastewater treatment. Most of these studies mainly focus on the design of aerators and optimizing operating factors for conventional aeration bubbling methods. These studies focused on methods to enhance aeration efficiency to improve biodegradation of organic matter. The main concern is the mass transfer rate of the DO, the key water quality parameter for defining aeration efficiency (Temesgen, Bui et al. 2017) . Some researchers have recently found that using micro/nano bubbling aeration has the potential to improve the mass transfer of DO. The process resulted in a faster transfer rate and greater DO durability when compared with the conventional macro-bubbling aeration. Some research results showed that the oxygen utilization rate and oxygen mass transfer coefficient in the MNB can be up to double that of conventional bubbling (Temesgen, Bui et al. 2017) . Additionally, MNBs have a much higher possibility to attach to surfaces of dirts and contaminants in water than conventional bubbles (Ahmadi, Khodadadi et al. 2014) . The MNBs on the surfaces of adjoining particles will tend to bring the two particles into contact, thus leading to the aggregation of contaminating particles (Basile 2015) . The aggregated particles can be more easily captured by conventional bubbles, which will provide enough buoyancy forces to elevate contaminating particles to the water surface. A number of recent experimental studies have reported a significant increase of the flotation rate of microparticles in the presence of micro/nanobubbles (Ahmadi, Khodadadi et al. 2014, Basile 2015, Zhou, Chen et al. 2016) . Aligning with the same objective as other similar studies, we propose using an MNB system instead of a conventional bubbling method to treat winery wastewater. The research will investigate the improvement in DO generation and mass transfer resulting from the MNB, which should lead to an increase the COD/BOD removal efficiency in the winery wastewater.

Technical Challenges

In the literature, a conventional aeration system for winery wastewater treatment needs high energy input and maintenance cost to generate bubbles with mechanical aerators or diffusers. Therefore, there always exists a balance between aeration efficiency and economic consideration. It is necessary to find an effective aeration method to provide sufficient oxygen to maintain a high COD/BOD removal efficiency while saving energy and cost.

Scientific Approach

Objectives

This research will utilize a micro/nano bubbling (MNB) aeration technology to enhance the oxygenation capability to investigate the ability of this technology to increase the removal efficiency of COD/BOD in winery wastewater. A pilot aerobic process system, including an aerobic tank equipped with MNB technology followed by a settling tank will be designed and built at CSU, Fresno to investigate how the MNB affects the aerobic process in winery wastewater treatment. An electrochemical model will be developed to simulate and predict the aerobic processes in the aeration tank and to optimize the MNB
system.

Scope of Work

To achieve the goals mentioned above, the research team consists of four faculty from Fresno State's Lyles College of Engineering and Jordan College of Agricultural Sciences and Technology will fulfill the following six (6) tasks:

1. Investigate the properties of water quality of winery wastewater in central California by using a pilot secondary treatment unit (an aerobic tank and a settling tank) to treat winery wastewater
2. Quantitatively differentiate the dissolved oxygen (DO) level and retention time in the aeration tank compared with the traditional aeration methods
3. Investigate the impact of the MNB aeration rate on COD/BOD removal efficiency
4. Simulate and predict the MNB aeration process in the aeration tank by using an electrochemical mathematical model
5. Explore and rank the impacting factors on the BOD removal efficiency (sensitivity analysis)
6. Optimize the MNB operating factors by maximizing aeration efficiency and minimizing the cost.

Method and Materials

This research is a combination of experiments and a mathematical model. Tasks 1, 2 and 3 will be accomplished through the experiments using batch experiments and a CSTR aerobic process unit. Then an electrochemical mathematical model will be developed to simulate the aerobic processes under various operating conditions. The model will be validated and calibrated by comparing the modeling results to the experimental data. The calibrated model will be used for future prediction, sensitivity analysis, and operation optimization. The detailed tasks are as follows,

Task 1. Investigate the properties of water quality of winery wastewater in Central California (Drs. Liu and Sun)

1.1 Investigation of winery wastewater properties in California (Dr. Sun)

Water is used throughout the winemaking process from crushing to bottles for temperature control, cleaning, sanitation, sterilization, and filter rinsing. Cleaning waste is the most significant contributor to wastewater because it is generated at every wine processing stage. Wastewater characteristics vary seasonally. During harvest season, winery wastewater is high in organic content, containing

predominantly sugars, followed by organic acids (acetic, tartaric, propionic), esters, and polyphenolic compounds, which come from grapes, yeast, additives, and fining agents. During the bottling season, winery wastewater contains less organic content but more inorganic composition such as sodium, potassium, calcium, and magnesium ions, which come from cleaners and sanitizers (Johnson and Mehrvar 2020) . Winey wastewater has high BOD and TSS content, which has unique characteristics that differ significantly from other food processing wastewaters (Mosse, Patti et al. 2011). Winey wastewater characteristics are influenced by climate and wine type produced. They vary from winery to winery and change seasonally, with the highest organic loads occurring during harvest season.

California is the largest wine producing state in the country. It accounts for 89% of all American wine. California planted 635,000 acres of wine grapes (2019) , and crushed totaled 3,548,623 tons of wine grape in 2020 (2021) . Recent studies have shown that the relative volumes of wastewater generated by different wineries averaged around 3–5 thousand liters per metric tonne of crushed grapes (Kumar 2006) . There were 3.5 million tons of grapes crushed in the 2020 season, suggesting that the total volume of wastewater generated by the California wine industry during this season was in the order of 11–18 billion liters.

In this research, wastewater will be collected from a commercial winery (Fresno State Winery). pH will be tested by a pH meter. The water samples will be pre-analysized by measuring several water quality parameters in the Viticulture and Enology Research Center lab before they are delivered to the treatment unit. A conductor will test EC. A dissolved oxygen probe will be used to test DO and to determine the biochemical oxygen demand (BOD)). Chemical oxygen demand (COD), nitrate, nitrite, phosphate, sulfate, chlorine will be measured by using a UV5 spectrophotometer (Mettler Toledo, Columbus, OH). Concentrations of cations (Na + , K + , NH 4 + , Mg 2+ , Na 2+) will be measured using microwave plasma atomic emission spectroscopy. The data will also be used to calculate the following metrics of wastewater quality (concentrations in mEq/L): sodium adsorption ratio (SAR), potassium adsorption ratio (PAR), monovalent cation ratio (MCAR), and cation ratio of structural stability (CROSS).

$SAR = Na^{+}/[(Ca^{2+} + Mg^{2+})/2]^{1/2}$

$PAR = K^{+}/[(Ca^{2+} + Mg^{2+})/2]^{1/2}$

$MCAR = (Na^{+} + K^{+})/[(Ca^{2+} + Mg^{2+})/2]^{1/2}$

$CROSS = (Na^{+} + 0.56K^{+})/[(Ca^{2+} + 0.6Mg^{2+})/2]^{1/2}$

1.2 Secondary wastewater treatment system study using a pilot CSTR

A typical activated sludge process consists of a pretreatment process (mainly screening and clarification), a secondary treatment system including an aeration tank (bioreactor) and a final clarifier for sedimentation, and excess sludge treatment for anaerobic treatment and dewatering process. Figure 1 shows a flowchart of a conventional activated sludge wastewater treatment plant. This research will focus on the secondary treatment system (the red-dashed box). A pilot aeration tank and a secondary clarifier will be built in room EE 187 of the Lyles College of Engineering. A CSTR will be designed and built to simulate the aeration tank. Air is sparged under pressure from the bottom to provide sufficient oxygen for microbes. The aeration efficiency of MNB will be compared with that of the conventional bubbling methods. The winery wastewater sample will be prepared from the lab (Viticulture and Enology Research Center room 118) and delivered to the water quality lab for further chemical constituent analysis. The main water quality parameters for this task include dissolved oxygen (DO), chemical oxygen demand (COD), pH, and electrical conductivity (EC). These parameters will be measured with a Hach DR6000 laboratory spectrophotometer in the water quality lab of LCOE.

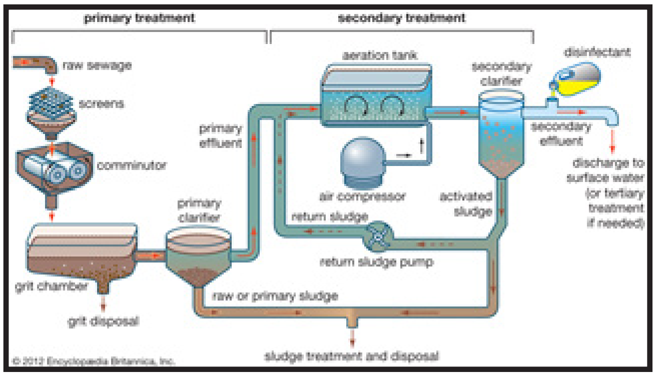

Figure 1. Flow chart of a conventional wastewater treatment plant(Ambulkar 2020)

Task 2. Quantitatively differentiate the dissolved oxygen (DO) level and retention time in the aeration tank compared with the traditional aeration methods (Dr. Liang)

Ordinary macrobubbles are unstable. They rise rapidly to liquid surfaces due to the buoyancy and then collapse. As the bubble size reduces, the terminal rise velocity of a spherical bubble quickly decreases. When the bubble size is less than 1 μm, the buoyancy effect on bubbles is insignificant compared to Brownian motion. As a result, these tiny bubbles, named micro-nanobubbles (MNBs) or ultrafine bubbles in the literature, can remain suspended in liquids for hours or even days (Alheshibri, Qian et al. 2016).

Using a variety of methods, including electrolysis (Kikuchi, Nagata et al. 2007, Kikuchi, Ioka et al. 2009), gas injection (Ohgaki, Khanh et al. 2010), agitation and cosmic radiation (Sette and Wanderlingh 1962, Seddon, Lohse et al. 2012), MNBs infused with air, O_2, O_3, or H_2 gases can be readily produced in water. As MNBs have a long lifetime, they have more time to deliver gas to the solution and to a larger surface area. Water MNB solutions have been demonstrated to have substantial positive effects on water treatment (Agarwal, Ng et al. 2011, Li, Hu et al. 2014), bioremediation of groundwater pollution (Li, Hu et al. 2014) (Li, Hu et al. 2014), surface cleaning (Yang and Duisterwinkel 2011, Zhu, An et al. 2016), and the growth of plants and fish (Ebina, Shi et al. 2013). In general, the production of MNBs has a low cost and the application of MNBs has a low environmental impact (Zhu, An et al. 2016).

We plan to purchase a commercialized nanobubble generator (from Moleaer Inc) which uses a special nozzle to generate micro/nanobubbles by gas injection. We will use the generator to conduct preliminary tests of efficiency of MNBs' used to treat winery wastewater.

Task 3. Investigate the impact of the MNB aeration rate on COD/BOD removal efficiency (Drs. Liang and Liu)

Operational factors of the aeration primarily include gas type (air or oxygen), gas flow rate through the nozzle of the MNB generator. COD/BOD removal is a function of time after the injection of MNBs. The removal efficiency RE of the contaminant i is defined as,

$$RE = \frac{\dot{M}_{in,i} - \dot{M}_{out,i}}{\dot{M}_{in,i}} \times 100\%$$

Where $M_{in,i}$ and $M_{out,i}$ are mass flowrate of species i in influent and effluent, respectively. The RE will be used to calculate the condition number, which is a parameter for quantifing the sensitivity of each operational parameter on the RE. In addition, some sensitivity analysis with the model in Task 4 will be conducted to study the indirect impact of the gas type and gas flowrate on other water quality parameters, including pH, EC, nitrate, ammonium. The impact on these parameters will be ranked according to their condition numbers. A higher condition number indicates higher sensitivity.

Task 4. Determine the aeration rate coefficient, simulate and predict the electrochemical processes in the aeration tank with the MNB aeration by using an electrochemical mathematical model (Dr. Xie)

Winery wastewater has variable acidity whose pH could varies from 3 to 11 because of the addition of acidic grape juice and alkaline cleaners (Mosse, Patti et al. 2011). The removal of high organic compounds and inorganic compounds in winery wastewater requires high efficiency oxygenation, which typically is a physicochemical process (Oliveira, Queda et al. 2009, Buelow, Steenwerth et al. 2015). Due to the stability and large contact surface of oxygen nanobubbles, the removal process of the organic and inorganic pollutant can be monitored electrochemically using a pair of electrodes. The physically adsorbed active oxygen (or adsorbed hydroxyl radicals) could cause the complete oxidation of organic compounds (Botte 2017, Akuzum 2019):

$$R + MO_x\,(^{\cdot}OH)_{ad} \rightarrow CO_2 + nH^+ + ne^- + MO_x$$

while the chemisorbed active oxygen participates in the formation of selective oxidation products that could lead to indirect oxidation reaction to destroy pollutants (Botte 2017, Akuzum 2019):

$$MO_x\,(^{\cdot}OH)_{ad} \rightarrow MO_{x+1} + H^+ + e^-$$
$$R + MO_{x+1} \rightarrow R + MO_x$$

The oxidation reaction rates can be formulated by,
where cRi and cPj are the concentrations of reactant and product respectively while ni and mj are the corresponding

stoichiometric factors; kf , kb are the forward and backward reaction rates respectively, and can be described by the

$$r = k_f \prod_i \left[c_{R_i}\right]^{n_i} - k_b \prod_i \left[c_{P_j}\right]^{m_j}$$

Arrhenius expression (Bessler, Gewies et al. 2007),

$$k_f = AT^n exp\left(-\frac{E_f^{act}}{RT}\right),\ k_b = AT^n exp\left(-\frac{E_b^{act}}{RT}\right)$$

where, A, n and E^{act} are the Arrhenius parameters for forward and backward reactions respectively. In the well-stirred aeration tank, the concentration of dissolved oxygen, c_{dis}, can be formulated by,

$$\frac{\partial c_{dis}}{\partial t} - \beta(c_{sat} - c_{dis}) = r$$

where, β is the rate constant of oxygen dissolving; c_{sat} is the saturation concentration of oxygen in the water. Based on the experimental setup, a Computational Fluid Dynamic (CFD) model can be developed to model and analyze the concentration distribution of oxygen nanobubbles. Modeling results will be benchmarked by experimental observations and measurements. Then, the intensity of direct and indirect oxidation reactions and the COB/BOD removal efficiency in the aeration tank will be investigated at different MNB aeration rates and different pH values.

Task 5. Explore and rank the impacting factors for the COD/BOD removal efficiency (sensitivity analysis) and Task 6. Optimize aeration operations with the MNB for the pilot treatment system

The performance of activated sludge treatment with the aerobic processes is affected by some operating factors and influent characteristics, including wastewater flow rate, BOD and COD, nutrient compositions (nitrogen and phosphorus), FOG (fat, oil, and greases), alkalinity, heavy metals, toxins, pH, temperature, and etc. Operating factors in the treatment are biomass concentration (mixed liquor volatile suspended solids concentration (MLVSS) and volatile suspended solids (VSS)), organic load, food to microorganisms ratio (F/M), dissolved oxygen (DO), sludge retention time (SRT), hydraulic retention time (HRT), sludge return ratio, and surface hydraulic flow load. SRT and DO are the most crucial control parameters among these factors and can significantly affect the COD/BOD removal efficiency. In this research, the relationship between the aeration rate and these operating parameters will be investigated. The sensitivity of the impact of the aeration rate on these parameters will be quantitatively ranked. The calibrated model will also be used to optimize the operational parameters to ensure that the DO in the aeration tank is maintained at a level above 2 mg/L with the most economic aeration practices.

Deliverables

The deliverables of this project include

1. A pilot secondary treatment system for winery wastewater
2. An electrochemical mathematical model that can be used to simulate and predict similar aerobic treatment processes
3. Standard procedure and protocol for the similar winery wastewater treatment
4. Application manual of the MNB operating factors
5. Publications (Journal papers and conference proceedings)

Schedule

Table 1. Schedule (month from start of project, 36 months in total)

Task	Start	Finish	Duration
Data Collection and literature review	0	3	6
(1) Investigate the properties of water quality of winery wastewater in central California	0	6	6
1.1 Investigation of winery wastewater properties in California	0 0	1 6	1 6
1.2 Secondary wastewater treatment system study using a pilot CSTR	0	6	
(2) Quantitatively differentiate the dissolved oxygen (DO) level and retention time in the aeration tank compared with the traditional aeration methods	7	15	8
(3) Investigate the impact of the MNB aeration rate on COD/BOD removal efficiency	7	36	29
(4) Determine the aeration rate coefficient, simulate and predict the electrochemical processes in the aeration tank with the MNB aeration by using an electrochemical model	7	24	8
(5) Explore and rank the impacting factors for the COD/BOD removal efficiency	24	36	8
(6) Optimize aeration operations with the MNB for the pilot treatment system	24	36	8
Preparation of final report and distribution of deliverables	33	36	3
Agencies meeting	0	36	TAB
Yearly water quality assessment using the most recent data	12	36	

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