



Institution	University of California, Riverside (Legal name: The Regents of the University of California)
Project Title	Recovery of Inorganic Phosphorus from Membrane Antiscalant in Reverse Osmosis Concentrate
Faculty Director	Dr. Haizhou Liu, haizhou@enr.ucr.edu
Student Manager	Tushar Jain, tjain001@ucr.edu
Project Strand	Global



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Recovery of Inorganic Phosphorus from Membrane Antiscalant in Reverse Osmosis Concentrate

Tushar Jain, tjain001@ucr.edu

Dr. Haizhou Liu, haizhou@enr.ucr.edu

Project Strand: Global

Project Overview:

Phosphorous is a valuable element that is essential as a fertilizer in agricultural and food industry. However, it has been speculated that the world phosphorus production from phosphate rock will inescapably decrease due to the depletion of its natural reserves. Reverse osmosis (RO) concentrate has a considerable level of organic phosphorous, mainly due to the application of phosphonate-based antiscalant to prevent scaling. High phosphorus content due to concentrate disposal can lead to eutrophication in receiving water bodies. Organic phosphonate compounds can mobilize potentially toxic metals in the receiving water. However, RO concentrate is typically discharged without recovering the phosphorous from organic phosphate in antiscalant. A hybrid adsorption-desorption-oxidation process is proposed in this study to concentrate phosphorus in its inorganic form on granular ferric hydroxide, recover it by subsequent desorption and convert it to valuable inorganic phosphorous minerals by chemical or enzymatic oxidation. Our preliminary results showed that essentially all phosphorus in antiscalant could be removed from the RO concentrate by adsorption. This unique process has a great potential for better desalination concentrate management, especially for inland desalination, reduce the effluent phosphorus concentration and produce valuable inorganic phosphate minerals simultaneously.

Contact Information

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3.A

Application Strand	Indicate Local or Global
LOCAL Project Name	
GLOBAL Project Name	X

3.B

Faculty Project Manager	Dr. Haizhou Liu	
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3.C

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3.E. PROJECT MANAGEMENT TEAM

Identify the team members of the project (i.e., budget, research, technology etc.). Add rows, as needed.

	NAME	TITLE / ORGANIZATION	ADDRESS	PHONE & EMAIL
1	Ms. Teeny Ellis	Senior Contract and Grant Officer (authorized representative)	Office of Research and Economic Development, 200 University Office Building, University of California, Riverside, CA 92521	951-827-2205 Teeny.ellis@ucr.edu
2				
3				

3.E. MEMBER AGENCY(IES) / LOCAL WATER AGENCY(IES)

	NAME	TITLE / ORGANIZATION	ADDRESS	PHONE & EMAIL
1	Tushar Jain	Graduate student		tjain001@ucr.edu
2	Changxu Ren	Undergraduate student		heyrex3@gmail.com
3	Dr. Haizhou Liu	Principal investigator		haizhou@enr.ucr.edu

4. Organizational Background

The Bourns College of Engineering (BCOE) at the University of California, Riverside, celebrated its 25th anniversary in 2015. The College now has more than 100 faculty members in nine degree programs: Bioengineering, Business Informatics, Chemical Engineering, Computer Engineering, Computer Science, Electrical Engineering, Environmental Engineering, Materials Science and Engineering, and Mechanical Engineering. The College has more than 2,000 undergraduate and graduate students. UCR is one of the 10 campuses of the University of California system, and one of America's most diverse research-intensive universities. We are an accredited Hispanic Serving Institution (HSI, OPEID-00131600).

This project will operate from the Department of Chemical and Environmental Engineering, where Professor Liu holds a faculty appointment and Mr. Jain is a graduate student. The Department offers leading-edge research and education in fields that will change the future of health, energy, public safety, and the quality of our air, water, and land. The Department and two closely affiliated research centers (the College of Engineering-Center for Environmental Research and Technology and the Center for Nanoscale Science and Engineering) are leading the way on overcoming some of the most challenging scientific and technological problems of our time. For example, our faculty and students are leaders in the development of innovative methods to control air pollution and emissions from transportation and industrial sources. We are developing technologies to assure abundant supplies of safe drinking water. We are applying nanoscience principles to the creation of new sensors that can detect toxic substances in air or water rapidly, accurately, and inexpensively. And we are advancing toward the development of clean and renewable fuels and energy that can provide for society's needs sustainably and economically.

As a research-intensive university, we understand that our role involves (1) educating the next generation of engineers for leadership in innovation to solve problems and satisfy needs, (2) producing innovations that lead to improved quality of life, environmental quality, and economic opportunity, and (3) performing service to the public. The *mission* of the Department of Chemical and Environmental Engineering is to prepare students for professional practice, graduate study, and life-long learning, and to advance the scientific and technological basis for chemical and environmental engineering practice.

Because of the rapidly changing technological society in which we live, today's chemical and environmental engineering graduates cannot be rooted into a single, standard mode of operation. They must be able to adapt readily to changing technologies and problem emphases, and develop creative solutions that are responsive to society as a whole. Thus, today's engineering students need to be rooted primarily in principles, not techniques. Opportunities to perform research are essential for us to educate our students. They require understanding of fundamental principles and an understanding of scientific methods and approaches. They build the ability to work in heterogeneous teams. They engender a sense of curiosity and empower rising engineers to pursue interesting lines of inquiry.

5. Certification of Attendance



Recovery of Phosphorus from Membrane Antiscalant in Reverse Osmosis Concentrate

Phosphorus is a non-metal that is never found as a free element in nature. It is present in the earth's crust in rocks, soils mainly as mineral salts of inorganic phosphates, and in water and living organisms. Phosphorus is primarily obtained in the form of phosphates from phosphate rocks for all commercial purposes and hence is the sole raw material. Major uses of phosphorus are to make fertilizers and phosphoric acid. Most of the phosphorus mined is used as synthetic fertilizer in agriculture. Normally, nitrogen, potassium and phosphorus are present in soil which are required as nutrients. However, they might not be present in a quantity adequate for plant growth. Hence phosphorus containing fertilizers are added to the soil to boost the productivity to the required levels. It cannot be substituted with any other element and is the key for all essential growths. The use of these fertilizers have been increasing continuously due to increasing food demand owing to increasing population.

Phosphate reserves are distributed across the globe based on its composition and quality. All the requirement of phosphate for fertilizer comes from naturally occurring phosphate rocks. Morocco is the largest exporter of phosphate rocks. United States, Russia, China and Morocco are the major producer of phosphate rocks. These countries contribute to almost 75% of the world output¹. It has been speculated that the world phosphorus production from phosphate rock will peak to its high in the decade of 2030-40 and then the production will inescapably decrease due to the depletion of its natural reserves. Florida is one of the leading producer of phosphorus in the United States. However a report suggested that the phosphate rock reserves in Florida could be totally depleted by 2050². Due to the shrinking supply and ever increasing demand the quality of reserves is declining³.

Moreover, the use of alternate water purifying technologies have increased due to ever-shrinking fresh water reserves, thereby limiting the availability from conventional fresh water resources. Desalination of ocean water is seen as one of the alternatives for supplying freshwater compared to other conventional processes. In membrane related desalination processes phosphorus-containing chemicals (i.e., organophosphates) are used as antiscalants to prevent scaling of membranes. These antiscalants have excellent chelating properties and are also very effective inhibitor of crystallization, preventing mineral precipitation, thus raising the saturation point of sparingly soluble salts including calcium carbonate (CaCO_3), calcium sulfate (CaSO_4) and others. It is estimated that the concentration of organic phosphorous in the RO concentrate can reach several mg/L. This is a significant source of phosphorous. However, RO concentrate is typically discharged without recovering the phosphorous from organic phosphate in antiscalant. It is estimated that the concentration of organic phosphorous in the RO concentrate can reach several mg/L. This is a significant source of phosphorous. However, RO concentrate is typically discharged without recovering the phosphorous from organic phosphate in antiscalant.

These phosphonates also contribute to the total phosphorus content in the effluent reverse osmosis concentrate and is considered to be a compound that promotes eutrophication of the receiving surface water. This leads to extensive growth of harmful algal blooms and depletion of dissolved oxygen that eventually results in the decline of marine life⁴. While nitrogen and carbon required for the algal growth are readily available from nature, phosphorus is a limiting reagent which is provided to the water bodies either by rainwater runoffs or by anthropogenic activities like agricultural water runoffs and industrial wastewater effluents. In addition, as excellent chelating agents, organic phosphonate compounds can mobilize potentially toxic metals in the receiving water. At a global scale since 1965 until 2005 the nitrogen loading has doubled, and the total phosphorus loading has tripled in water bodies. It is a problem that is faced globally and more severely in the sub-tropical region. Based on the a survey⁶, on the extent of problems faced on a global level has found that in Africa, South East Asia, North and South America, 28%, 54%, 48% and 41% respectively of the lakes and reservoirs are eutrophic. Whereas, 53% of the lakes in Europe are eutrophic.

Over the last few decades the regulations regarding the contribution of phosphorus from industry, including municipal wastewater treatment facilities, have been continuously tightened in efforts to prevent the potential impacts associated with excess phosphorus, as a required nutrient, to the environment. The RO brine can concentrate nutrients which can be removed by advanced oxidation processes. Recovery of phosphorus in its organic form as an antiscalant is economically less feasible due to the downstream processing required to extract the antiscalant in its applicable form. Also, it is important and at the same time difficult to maintain the structure of the complex organic antiscalant throughout the process for it to retain its chelating properties. In this project, we proposed an idea to recover the phosphorus in its more valuable inorganic form which can be used as a fertilizer. Currently, little research has been done to investigate the potential to extract organic phosphonate from RO concentrate, convert it to inorganic phosphorous and recover as a valuable phosphate fertilizer. Technologies for removing phosphorus from the concentrate are still unexplored. Recovering the phosphonate in its antiscalant form has received some attention in the recent years⁵. However, no prior research has investigated the recovery of phosphate as an inorganic fertilizer.

In this project, we proposed a ground-breaking approach to achieve phosphorous recovery from RO concentrate. Our proposed approach of innovation is to include a quaternary step in the treatment of wastewater that would concentrate the brine (or the concentrate) by a hybrid absorption-desorption-oxidation technique with high inorganic phosphorus concentration. The entire process is a simple adsorption of organic phosphonate on the surface of granular ferric hydroxide (GFH) from RO concentrate, subsequent desorption of the organic phosphonate from GFH at elevated pH to generate a concentrated solution. Following that, an advanced oxidation step using ultraviolet-based hydrogen peroxide (H_2O_2) employed to convert organic phosphonate to valuable inorganic phosphorous mineral as fertilizer. This would help overcome

the restriction barrier of the MCL for phosphorus and generate an in-demand sellable by-product in terms of a nutrient for fertilizer.

In order to remove phosphorus from wastewater effluents, treatment facilities often apply removal technologies that involve chemical methods such as aluminum or iron salts or using an activated sludge treatment that involves biological phosphorus removal, or a combination of both. However, chemical method is capable only of removing orthophosphate. Thus, organic phosphates and polyphosphates easily escape through this process. GFH is known to be an excellent adsorbent with a higher capacity of adsorption for phosphorus from water due to the strong binding properties⁷. It has been successfully used as an adsorbent for arsenic removal from wastewater. This affinity of phosphorus to the surface of GFH gives it an edge over adsorption of other anions (Geelhoed et al., 1997 and Genz, 2005), mainly due to interactions of surface charges. GFH has a point of zero charge (pzc) near a pH of 8.8. When the solution pH is below this pzc value, the surface is positively charged and negatively charged on the other side. This property of GFH can be exploited to facilitate adsorption followed by desorption just by altering the solution pH. To recover the phosphorus in its inorganic form we used an advanced oxidation process which involves using a polychromatic ultraviolet lamp along with hydroxyl radicals as an oxidizing agent supplied by hydrogen peroxide.

Nitrilotrismethylene phosphonic acid (NTMP) was used as a model antiscalant compound as an adsorbate for all the experiments. The concentration of NTMP varied from 0.5 to 2 mg-P/L. GFH was used as the adsorbent for all the experiments with its concentration varied in the range of 0.5 to 2 g adsorbate per liter. The pH of the solution adjusted by using concentrated perchloric acid and sodium hydroxide. The solution was oxidized by using a polychromatic UV lamp with hydrogen peroxide as the oxidizing agent for 60 minutes.

Experiments were carried out in a batch mode. The pH of Reverse osmosis concentrate is slightly basic, close to 7.8. At this pH, the GFH surface is positively charged and has a potential to adsorb negatively charged phosphonate group. The solution is stirred uniformly for 4 hours at 400 RPM and allowed to reach equilibrium. The adsorbate (GFH) is allowed to settle down and made to bind to the bottom by centrifuging it at 4000 RPM for 40 minutes. The solution is then concentrated to 16 times by decantation. Samples for supernatant are collected for analysis of remaining phosphorus in the solution. The pH for the concentrate solution is then raised to a desorption pH in the range of 9 to 11 and stirred at 400 RPM for 150 minutes. The concentrate is centrifuged again for 40 minutes at 4000 RPM. The photochemical oxidation experiment was carried out for 60 minutes at a wavelength spectrum of 200-600nm using a medium pressure UV lamp to convert organic phosphonates to simple inorganic phosphate. A sample from the supernatant of the concentrated solution is taken for analysis of total adsorbed phosphorus in the solution. The total adsorbed and desorbed phosphorus is quantified using ascorbic acid method by measuring the absorbance via UV/Vis spectroscopy according to Beer-Lambert law.

The research objectives are to:

- (1) Examine the time dependence of adsorption of organic phosphonate on GFH
- (2) Examine the impact of ions in RO concentrate on adsorption of organic phosphonate on GFH, for example, calcium (Ca^{2+}) since it is present in high concentrations in RO concentrate.
- (3) Optimize the desorption of organic phosphonate and its conversion to inorganic phosphorous during the separation processes.

Progress to date:

At the experimental conditions mentioned above, the impacts of adsorption time (i.e. 4 and 24 hours) and the presence of cation (i.e., Calcium) on phosphorous recovery are shown in Fig 1. Experiments were carried out with varying concentrations of calcium. Based on the conditions tested so far, a maximum recovery of 37% of antiscalant has been achieved at a molar phosphorus: calcium ratio of 1:2 with a 24 hours' adsorption experiment. However, the presence of calcium does not show a very significant increase in the recovery. The recovery obtained with 24 hours' adsorption experiment is much higher than the 4 hours' adsorption, suggesting that the recovery percentage depends on adsorption time.

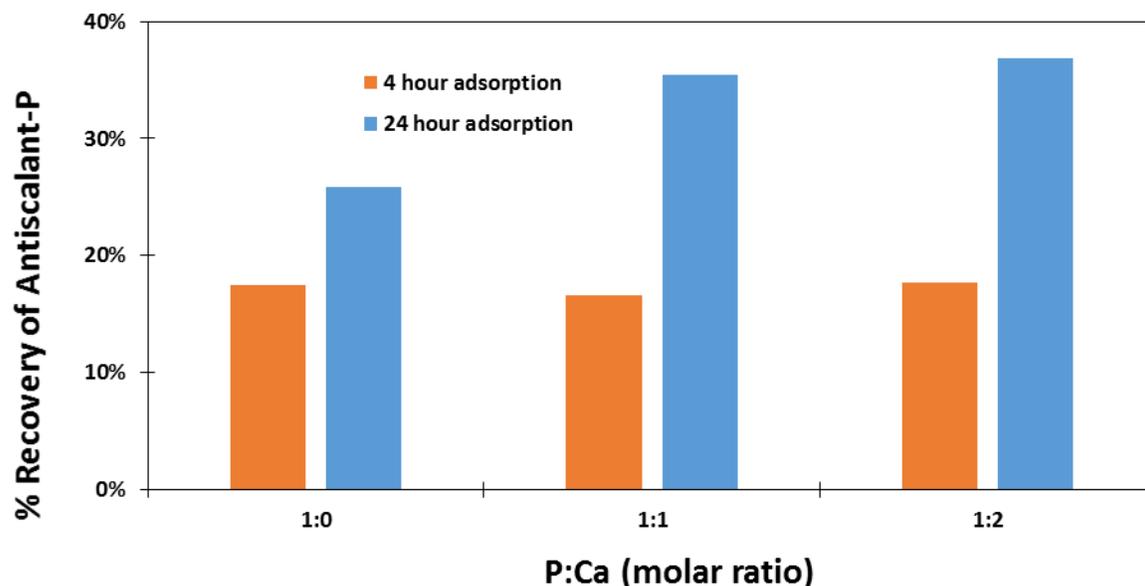


Figure 1: Impact of P-to-Ca molar ratio on the efficiency of recovery of phosphorus for various [P:Ca] ratio as antiscalant. Initial pH: 7.8, GFH: 2.6 g/L. antiscalant: 1.5 mg-P/L. Batch volume: 250 mL

During the recovery process, the distribution of the antiscalant as phosphorus can be divided into three groups:

- (i) Unrecovered antiscalant from the bulk RO concentrate (i.e. remained in the solution)
- (ii) Extracted from the bulk RO concentrate and recovered from the adsorbent (i.e. recovered)

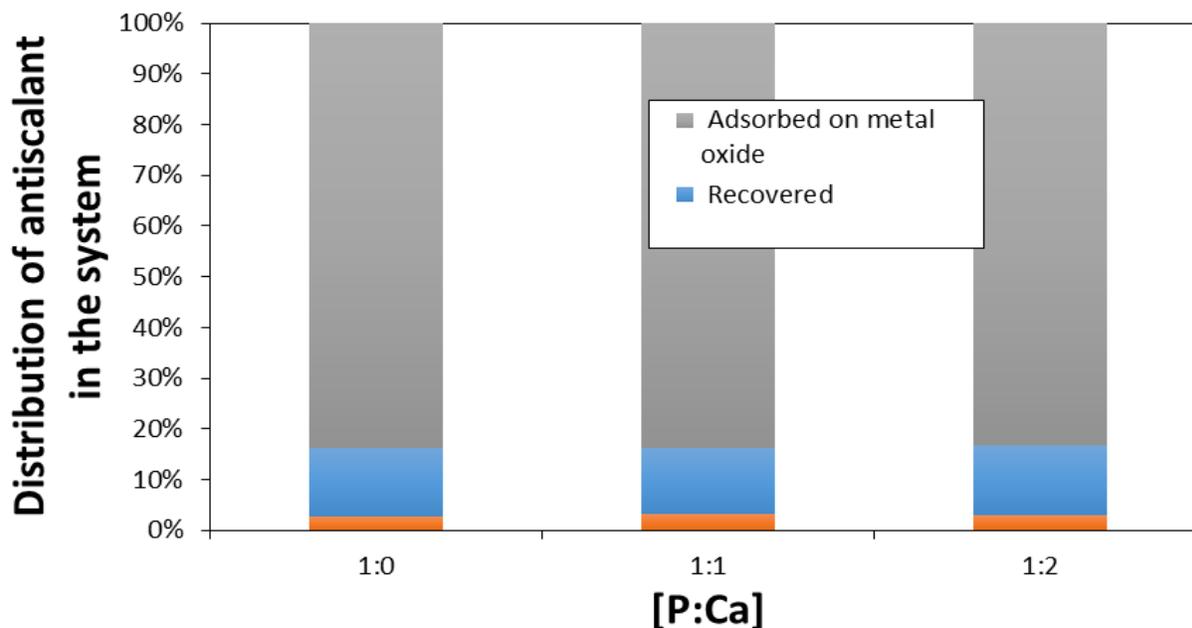


Figure 2: Distribution of antiscalant in the separation process for different P-to-Ca molar ratios. Initial pH: 7.8, GFH: 2.6 g/L. antiscalant: 1.5 mg-P/L. Batch volume: 250 mL. Batch time: 4 hours.

(iii) Antiscalant that is recovered from the bulk RO Concentrate but could not be desorbed from the adsorbent (i.e. adsorbed on metal oxide)

Figure 2 shows the antiscalant distribution in the end of the process. It is evident from the distribution graph that more than 97% of the antiscalant was removed from the RO concentrate thus bringing down the concentration of phosphorus in the antiscalant from 1500 $\mu\text{g P/L}$ to 40 $\mu\text{g P/L}$. Of the 97% removed, 20% of the antiscalant could be recovered from the solution. Thus, a major portion of antiscalant remains unaccounted for. The possible reason for that could be an inefficient desorption process which led to only a partial recovery of the antiscalant from the GFH.

Another reason could be incomplete transformation of organophosphate to inorganic phosphate. The analysis method relies on measurement on the transformed organophosphate to inorganic phosphate. However, the later hypothesis seems less contributable due to the fact that the increase in the hydrogen peroxide dosage in the UV-photolysis step for conversion of organic to inorganic phosphate did not change the measured results.

Future plans:

The results indicate that a reasonable removal of antiscalant (final concentration of 40 $\mu\text{g-P/L}$) can be achieved by this process. Thus this can prove to be a promising technique for removing

antiscalant (phosphorus) from the reverse osmosis concentrate. However, the downstream process still needs optimization to improve the recovery of the removed phosphorus.

Task 1:

To increase the ionic strength of the system by adding concentrated NaCl. The Bowden effect⁸ emphasizes the impact of ionic strength on the electrostatic potential developed at the surface of the adsorbent. The distribution of charge on the surface changes with change in the concentration of the electrolyte in the system. If the potential at the surface changes because of change in the electrolyte concentration and consequent change in the distribution of charge, the activity of the ion in the solution must also change in order to maintain the same activity of the adsorbed ion at the surface and thus the same amount of adsorption. Increasing the ionic strength can suppress the double layer and hence can reduce the bond between the adsorbed species. Reverse osmosis concentrate also has a chloride concentration of around 500 mg/L. Different dosage of NaCl will be investigated.

Task 2:

Study the effect of presence of other species such as Mg^{2+} and SO_4^{2-} . Similar to Calcium, other entities may or may not have an effect on the recovery process. Solid surfaces are always to some extent hydrated in aqueous solutions, and it has long been known that the structure of the interfacial water is fundamentally different from that in the bulk^{9,10}. Therefore, it is reasonable to assume that the presence of structure-breaking or structure-making ions at the mineral–water interface will additionally affect/alter the interfacial water structure, and hence influence the adsorption. Thus trying these species with typical concentrations found in the RO concentrate (41 mg/L Mg^{2+} and 268 mg/L SO_4^{2-}) will give an insight to the extent of removal based on realistic conditions.

The potential application of this innovative hybrid technology on RO concentrate as a quaternary treatment for effective reduction and recovery of phosphorus from effluent wastewater can provide an insight to the long term management strategy for the eutrophication related problems and also provide an alternate source of phosphorus in the fertilizer industry.

Reference:

1. Brentnall B., “Phosphate fertilizers”, Ullmann's Encyclopedia of Industrial Chemistry, Electronic Release, chap. 5, Wiley-VCH, Weinheim September 2005.
2. Jasinski, S. Phosphate Rock Reserves in the United States, Paper presented at the 2005 IFA Production and International Trade Conference, Sao Paolo, Brazil, September 11-14, 2005.

3. Runge-Metzger, A., Closing the cycle: obstacles to efficient P management for improved global food security. SCOPE 54 – Phosphorus in the Global Environment – Transfers, Cycles and Management. 1995.
4. A.M. Farmer. Phosphate pollution: a global overview of the problem. In E. Valsami-Jones, editor, Phosphorus in Environmental Technology. IWA Publishing, London, 2004.
5. Mohammadesmaeili, F.; Badr, M.; Abbaszadegan, M.; Fox, P. Mineral recovery from inland reverse osmosis concentrate using isothermal evaporation, Water Research, 2010, (44) 6021-6030.
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7. Cornell, R.; and Schwertmann, U. The iron oxides: structure, properties, reactions, occurrences, and uses; Wiley-VCH, Weinheim, 2nd, completely rev. and extended edition, 2003
8. Shainberg I., 1966, Hydration status of adsorbed cations. *Ibid.*, (30), 703-13.
9. Horne A., Day A., Young R., Yu N., *Electrochim. Acta*, (13), 1968, 397.
10. Franks F., *Water: A Comprehensive Treatise*, Plenum, New York, 1972

Quantitative Benefit Projections

PERFORMANCE MEASURE	QUANTITATIVE OUTCOME	LOCAL / GLOBAL IMPACT
Makes More Water Available	Acre Feet/Year	Global
Reduces Water Treatment Costs	\$ / Year	Local
Reduces Per Capita Use	Gallons/Capita/Day	Local
Provides Technical Training	# of People	Local / Global
Provides Water Conservation and / or Hygiene/Public Health Education	# of People/Students	Local / Global
Improves equitable access to fresh drinking water and/or sanitation practices (<i>e.g. by improving water quality</i>)	100,000*	Local
Improves the environment and sustainability benefits for people (<i>e.g.- by improving watershed runoff</i>)	100,000*	Local
Cost associated with each of the physical quantitative outcomes above	\$/person, \$/AF/yr and Gallons/Capita/Day	Local/Global

*Assuming that an average population of a town/city surrounded by a water body is approx. 100,000 on a local scale.

Matching Funds

The University proposes a match of 25% of the amount requested (i.e., \$2,500) in the form of volunteer time. We estimate that undergraduate students participating in this project will contribute more than 200 hours of effort, and we will track this activity. Student effort is valued at \$12 per hour or more.

BUDGET OVERVIEW

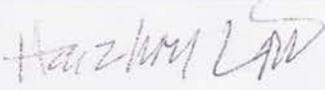
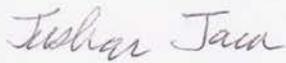
DESCRIPTION	AMOUNT	NOTES
GRANT FUNDS REQUESTED	\$9,978	
ADDITIONAL SOURCE OF FUNDS (List all, if applicable)	\$2,500	DATE ISSUED (If applicable) Not applicable – see paragraph above.
PROJECT TOTAL	\$12,478	

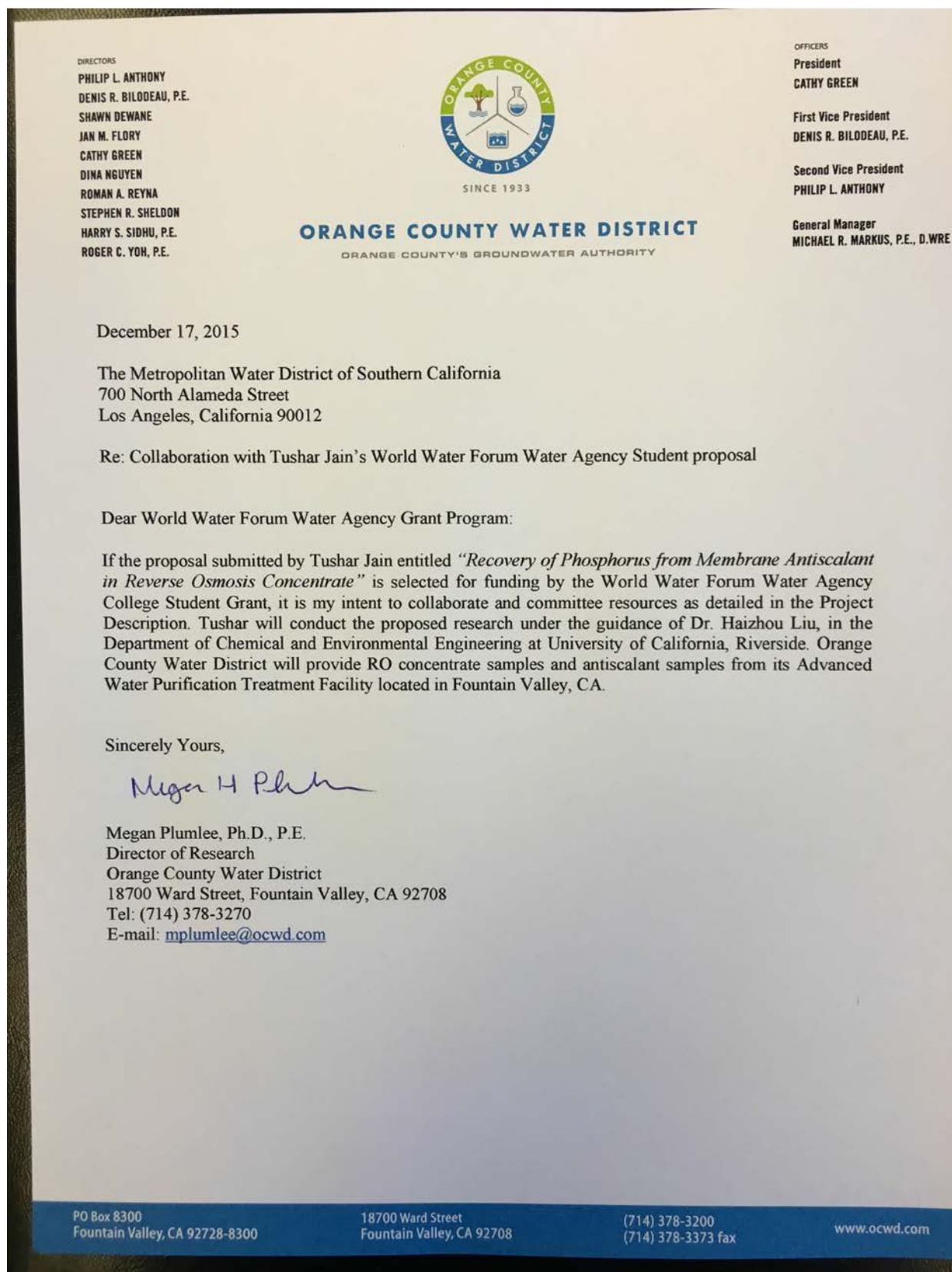
BUDGET DETAIL

A detailed budget is provided on the following page.

DIRECT COSTS						
	COMPUTATION					
BUDGET ITEM DESCRIPTION	PRICE/RATE	UNIT	QTY	MWD/ WWF	College	TOTAL COST
SALARIES AND WAGES						
Undergraduate Student Researchers				\$0	\$2,500	\$2,500
Graduate Student Researcher				\$7,571		
Subtotal			Subtotal	\$7,571	\$2,500	\$2,500
TRAVEL						
Airfare\Lodging (not allowed)						
Subtotal	2500		Subtotal	0	0	\$ -
SUPPLIES/MATERIALS - Describe all major types of supplies/materials, unit price, # of units, etc., to be used on this assisted activity.						
Misc. Chemical Cost	\$500		1	\$500		
Glass Ware	\$500		1	\$500		
Gases and Sample Analysis Cost	\$500		1	\$500		
Subtotal			Subtotal	\$1,500	\$0	\$0
CONTRACTUAL/ CONSTRUCTION						
Subtotal			Subtotal	\$0	\$0	\$0
TOTAL DIRECT COSTS:				\$9,071	\$2,500	\$2,500
INDIRECT COSTS -						
Indirect Costs @ 10% total				\$907	\$0	\$907
Subtotal				\$907	\$0	\$907
TOTAL INDIRECT COSTS				\$907	\$0	\$907
TOTAL ESTIMATED PROJECT/ACTIVITY COSTS:				\$9,978	\$2,500	\$12,478

8. SIGNATURE BLOCK

	NAME / TITLE	SIGNATURE	DATE
Faculty Project Manager	Dr. Haizhou Liu Assistant Professor		12/16/2015
College Contracts Officer / Administrator	Ms. Teeny Ellis Senior Contract and Grant Officer		12/17/2015
Student Project Manager	Mr. Tushar Jain Ph.D. Student		12/16/2015



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ORANGE COUNTY WATER DISTRICT
 ORANGE COUNTY'S GROUNDWATER AUTHORITY

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MICHAEL R. MARKUS, P.E., D.WRE

December 17, 2015

The Metropolitan Water District of Southern California
 700 North Alameda Street
 Los Angeles, California 90012

Re: Collaboration with Tushar Jain's World Water Forum Water Agency Student proposal

Dear World Water Forum Water Agency Grant Program:

If the proposal submitted by Tushar Jain entitled "*Recovery of Phosphorus from Membrane Antiscalant in Reverse Osmosis Concentrate*" is selected for funding by the World Water Forum Water Agency College Student Grant, it is my intent to collaborate and committee resources as detailed in the Project Description. Tushar will conduct the proposed research under the guidance of Dr. Haizhou Liu, in the Department of Chemical and Environmental Engineering at University of California, Riverside. Orange County Water District will provide RO concentrate samples and antiscalant samples from its Advanced Water Purification Treatment Facility located in Fountain Valley, CA.

Sincerely Yours,

Megan Plumlee, Ph.D., P.E.
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