2020-2022 Goals and Accomplishments

Strategic Program Area (1) - Produced Water Characterization

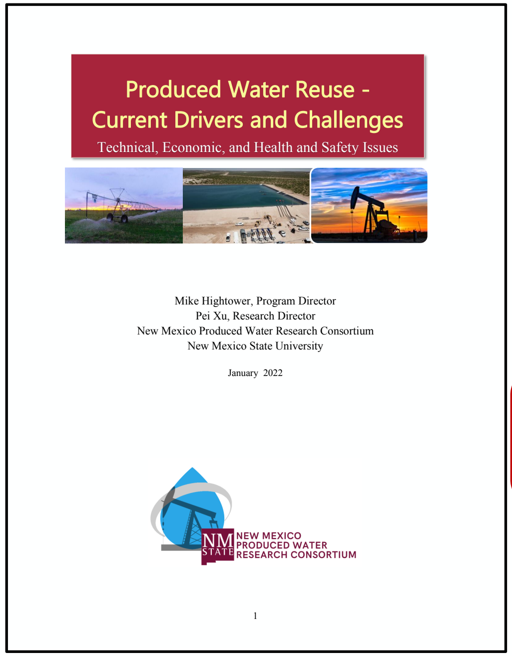
Pei Xu, Research Director

College of Engineering

2022 ANNUAL REVIEW

NM STATE NEW MEXICO PRODUCED WATER RESEARCH CONSORTIUM

2019-2020 Consortium Produced Water Reuse Gap Analysis

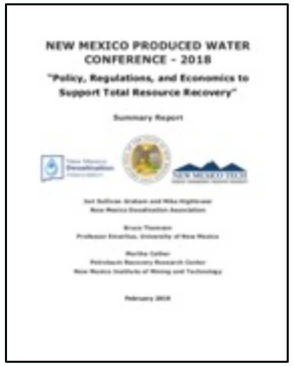


2019-2021

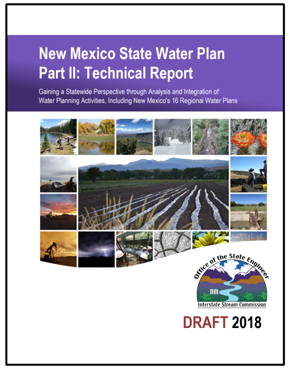
Priority Research Needs Identified

- Treatment to reduce fresh water use (6/6)
- Treatment cost/performance data (5/6)
- Develop treatment standards (5/6)
- Risk-based human and environment health and safety model (5/6)
- **Produced water characterization (6/6)**
- GIS data portal of volume and quality (4/6)
- Quantitative ESG model of costs vs benefits
- Infrastructure Implementation strategy

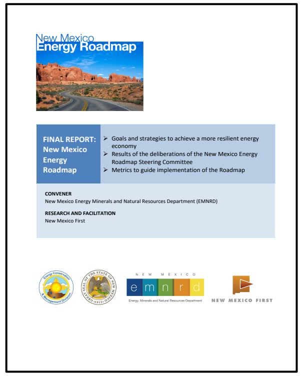
2016-2018



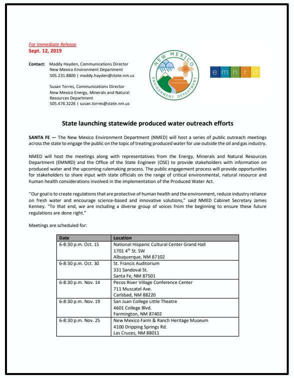
2018



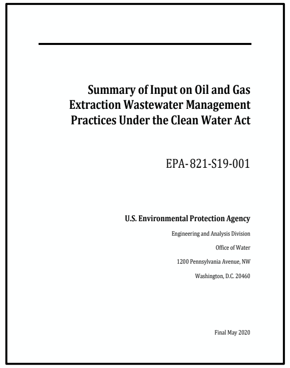
2016-2018



2017-2019



2019



2019-2020

Produced Water Characterization is Challenging

- Constituents of concern in produced water (formation water and flowback water):
  - Suspended solids, oils, and grease
  - Salts (referred to as dissolved solids) and metals
  - Dissolved organics (e.g., petroleum hydrocarbons, volatile and semi-volatile compounds)
  - Dissolved gases (e.g., H_2S, NH_3)
  - Naturally occurring radioactive material (NORM)
  - Microorganisms
  - Chemical additives (well completion and on-going well maintenance)
  - Transformation/degradation products, and unknowns
- Produced water quality and quantity are highly variable, spatial and temporal
- High salinity and complex water chemistry cause challenges in analytical methods and treatment
- Costly and time-consuming for “comprehensive” analysis

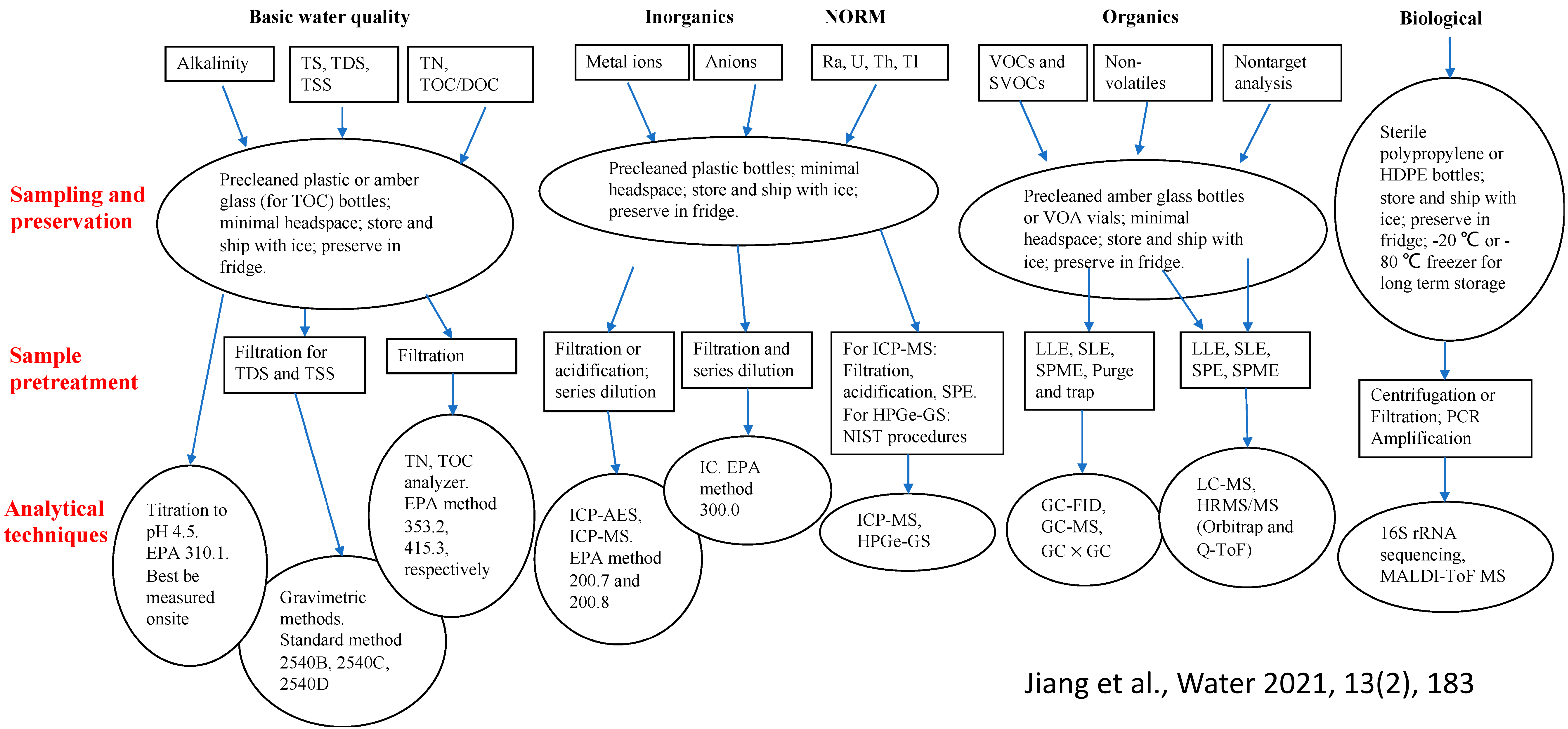
Highlights of Accomplishment

Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water

- No standardized methods approved by EPA on PW analysis
- Many studies rely on methods originally designed for surface and groundwater matrices. Flowback and PW have more complex matrices and higher salinity.
- Limited knowledge regarding the composition of chemical additives used during hydraulic fracturing, and the transformation products are unpredictable.
- The contaminants are usually present in trace amounts. To analyze these chemicals, sophisticated analytical methodologies are often required.
- Danforth et al. identified 1198 unique chemical constituents in PW, and only 290 (24%) could be quantified by the EPA-approved test methods
- The development of suitable analytical methods for accurate PW characterization in complex water matrices is imperative.

Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water

- Reviewed >150 peer-reviewed publications and regulatory standard methods.



Jiang et al., Water 2021, 13(2), 183

Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water

- Published in Water 2021, 13(2), 183; https://doi.org/10.3390/w13020183

water

MDPI

Review

A Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water

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Abstract: Produced water is the largest waste stream associated with oil and gas production. It has a complex matrix composed of native constituents from geologic formation, chemical additives from fracturing fluids, and ubiquitous bacteria. Characterization of produced water is critical to monitor field operation, control processes, evaluate appropriate management practices and treatment effectiveness, and assess potential risks to public health and environment during the use of treated water. There is a limited understanding of produced water composition due to the inherent complexity and lack of reliable and standardized analytical methods. A comprehensive description of current analytical techniques for produced water characterization, including both standard and research methods, is discussed in this review. Multi-tiered analytical procedures are proposed, including field sampling; sample preservation; pretreatment techniques; basic water quality measurements; organic, inorganic, and radioactive materials analysis; and biological characterization. The challenges, knowledge gaps, and research needs for developing advanced analytical methods for produced water characterization, including target and nontarget analyses of unknown chemicals, are discussed.

Keywords: produced water; water quality; hydraulic fracturing; analytical methods; treatment; reuse

check for updates

Citation: Jiang, W.; Lin, L.; Xu, X.; Cheng, X.; Zhang, Y.; Hall, R.; Xu, P. A Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water. *Water* **2021**, *13*, 183. https://doi.org/10.3390/w13020183

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1. Introduction

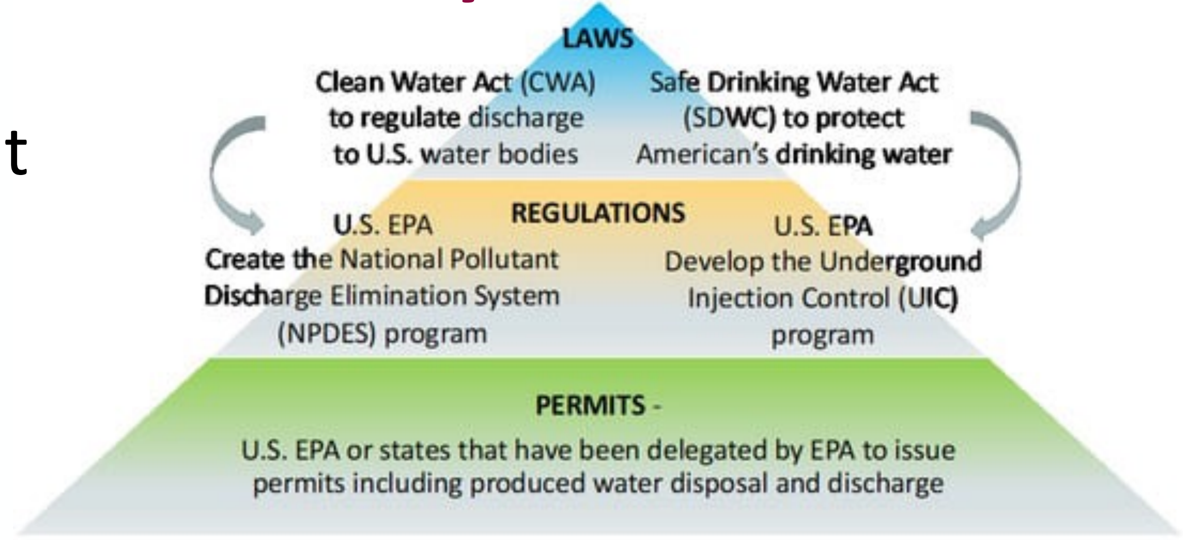
In 2020, the United States became a net energy exporter and will remain so until 2050, according to the U.S. Energy Information Administration [1]. Water resource management significantly influences the oil and gas (O&G) industry because water is used for almost all stages in fossil fuel production, such as well drilling and completion, reservoir management, enhanced oil recovery, and hydraulic fracturing (HF) [2]. HF uses a large volume of water to extract O&G from an "unconventional play" (or "tight oil play"), which refers to the low permeable unconventional shale that cannot be explored and produced by conventional processes relying on the natural pressure of the wells and pumping operation [3,4].

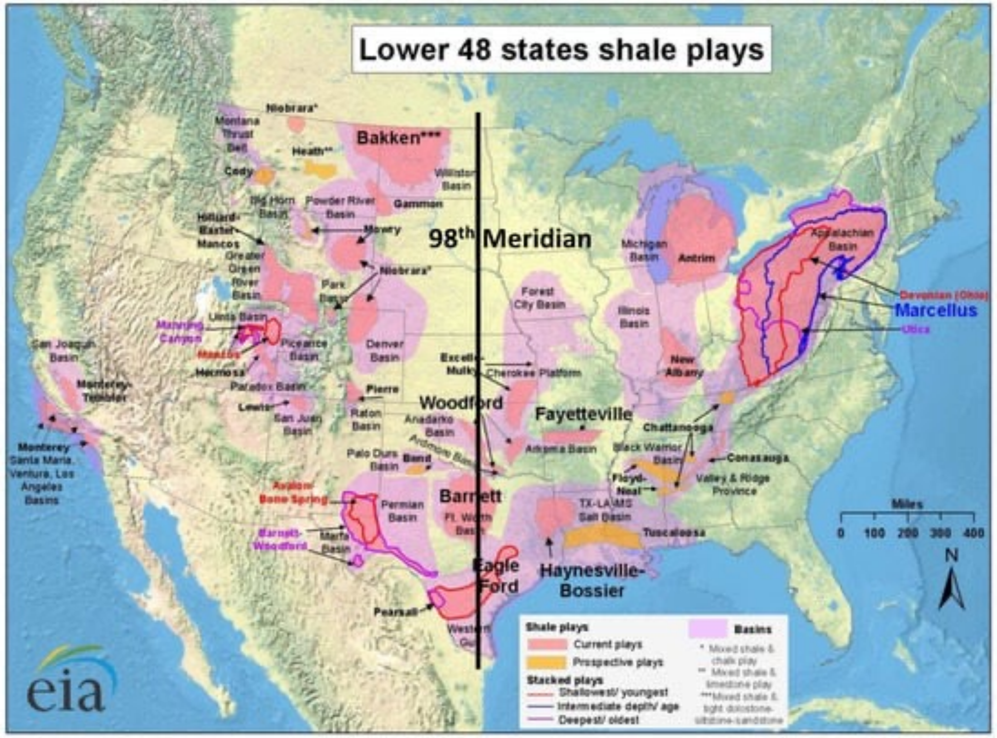
At the initial stage of HF, fracturing fluids are injected into deep wells under high pressure to fracture the geological formation, increase permeability, and extract oil and gas. Around 91–94% (mass percentage) of the fracturing fluid is water, with ~5–8% proppant (mostly sand) and ~1% chemical additives [5,6]. After HF, a portion of injected water returns to the surface with high levels of dissolved solids, salts, and chemical additives; this water is often referred to as flowback water (FW). FW usually occurs in the first several weeks and before the well is placed in production. Over time, FW diminishes and is replaced with formation water native to the well, which is referred to as produced water (PW), occurring throughout the life of a well [7]. In the field, FW and PW are commonly co-mingled so that these streams cannot practically be distinguished. Thus, PW is often broadly defined to include both water streams.

The United States produces an estimated 900 billion gallons of produced water (PW) annually, making it the largest waste stream associated with O&G activity [8]. The amount

What Constituents Should We Analyze?

- Case studies on regulatory framework, water policy, produced water management and reuse, and water quality standards
  - Pennsylvania, Ohio and West Virginia
  - Colorado
  - Texas
  - California
  - Oklahoma
  - Wyoming
  - New Mexico
- Literature review on beneficial use water quality requirements





Jiang et al., Water 2022, 14(14), 2162

California

- **Produced Water Quality Standards**
  - Discharge to land: cannot exceed the Basin Plan's maximum salinity limits for electrical conductivity (1000 µmhos/cm), Cl (200 mg/L), B (1 mg/L).
  - Recommended Irrigation Water Risk-Based Comparison (RBC) Levels (mg/L).

Inorganics		**Organics**	
Arsenic	0.1	Acetone	20,000
Barium	2,000	Benzene	0.7
Boron	70	Ethylbenzene	6
Cadmium	70	Ethylene Glycol	5,000
Chromium (VI)	0.4	Methylene Chloride	2
Fluoride	700	Naphthalene	200
Mercury	20	PAHs	0.02
Thallium	10	Toluene	500
Zinc	2,000	Total Petroleum Hydrocarbons	200
		Trimethylbenzene	200
		Xylenes	1,000

Constituent	Limit
Aluminum (mg/L)	0.2
Ammonia (mg/L)	2
Arsenic (ug/L)	10
Barium (mg/L)	2
Benzene (ug/L)	0.12
Beryllium (ug/L)	4
Boron (mg/L)	1.6
Bromide (mg/L)	0.1
Butoxyethanol (mg/L)	0.7
Cadmium (ug/L)	0.16
Chloride (mg/L)	25
Chromium (ug/L)	10
COD (mg/L)	15
Copper(ug/L)	5
Ethylene glycol (ug/L)	13
Gross Alpha (pCi/L)	15
Gross Beta (pCi/L)	1000
Iron (mg/L)	0.3
Lead (ug/L)	1.3
Magnesium (mg/L)	10
Manganese (mg/L)	0.2
MBAS (surfactants)	0.5
Methanol (mg/L)	3.5
Molybdenum (mg/L)	0.21
Nickel (mg/L)	0.03
Nitrite-nitrate nitrogen (mg/L)	2
Oil & grease (mg/L)	ND
pH	6.5-8.5
Radium (pCi/L)	5
Selenium (ug/L)	4.6
Silver (ug/L)	1.2
Sodium (mg/L)	25
Strontium (mg/L)	4.2
Sulfate (mg/L)	25
TDS (mg/L)	500
Toluene (Methylbenzene) (mg/L)	0.33
TSS (mg/L)	45
Uranium (ug/L)	30
Zinc (mg/L)	0.065

General Permit WMGR123 - Processing and Beneficial Use of Oil and Gas Liquid Waste, Pennsylvania Department of Environmental Protection.

Wyoming

Surface water discharge criteria:

- General guidance: "good enough quality" for watering livestock or wildlife.
- Effluent limits:
  - Chloride 2,000 mg/L,
  - Sulfate 3,000 mg/L,
  - Specific conductance 7,500 µS/cm,
  - Oil and grease 10 mg/L,
  - pH 6.5-9.0
  - Total recoverable radium 226 of 60 pCi/L

Groundwater injection criteria:

Injected into shallow aquifers bearing TDS<10,000 mg/L or <5,000 mg/L through Class V wells by mineral developers.

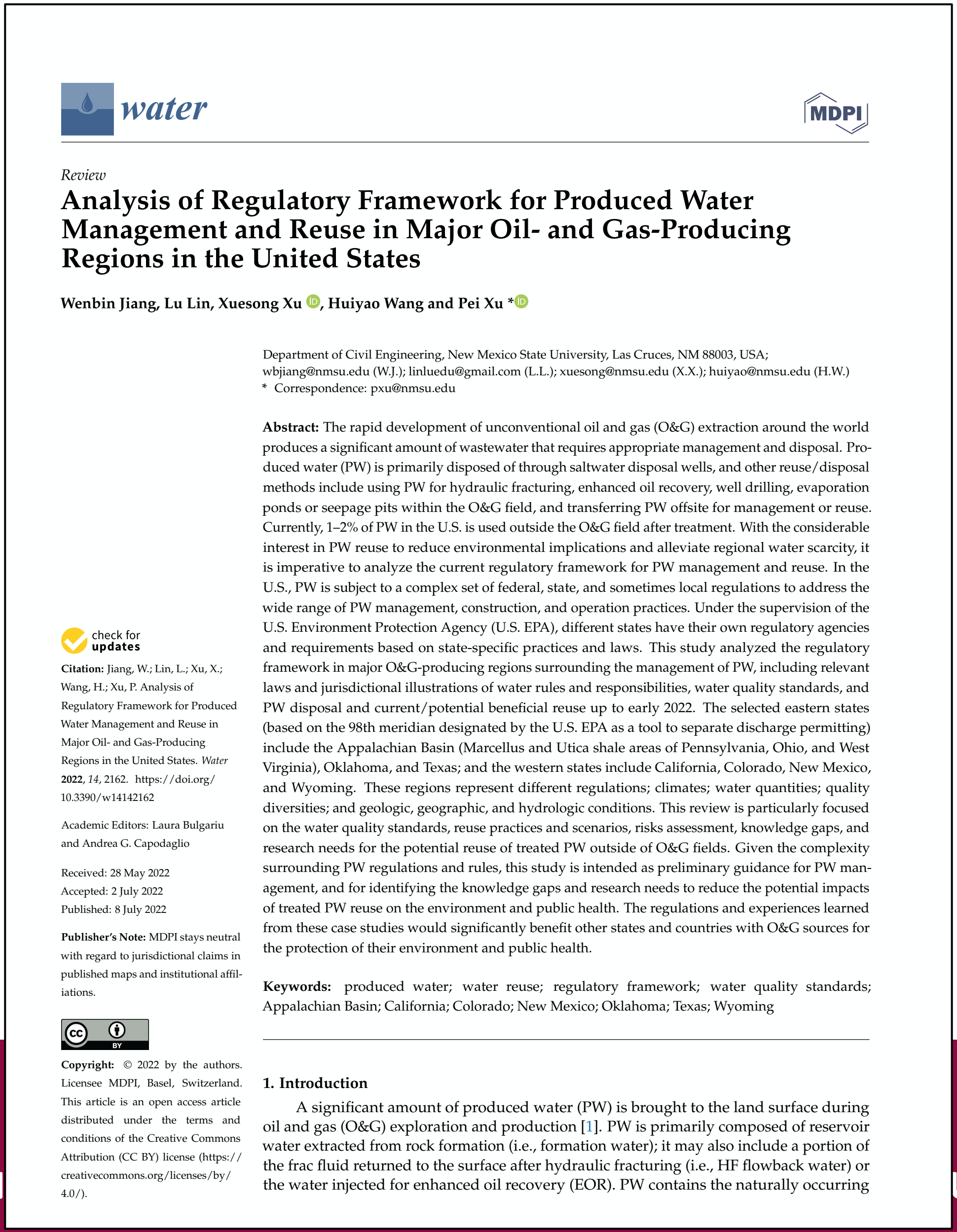
Wyoming

CBM produced water general quality criteria for land application in Wyoming.

	Limit		Limit		Limit
pH	4.5-9	Be (mg/L)	0.1	Hg (µg/L)	2
TDS (mg/L)	480	B (mg/L)	0.6	Mo (mg/L)	0.2
EC (µS/cm)	750	Cd (mg/L)	0.01	Ni (mg/L)	0.2
HCO_3 (mg/L)	<50% Total anion	Cr (mg/L)	0.1	NO_3-N (mg/L)	10
SAR	8-10	Co (mg/L)	0.05	NO_2-N (mg/L)	1
Al (mg/L)	5	Cu (mg/L)	0.2	Total-N (mg/L)	10
Sb (mg/L)	0.006	CN (mg/L)	0.2	H_2S (µg/L)	4.2
Ba (mg/L)	2	F (mg/L)	4	Se (mg/L)	0.02
As (mg/L)	0.01	Fe (mg/L)	5	Ag (mg/L)	0.2
Cl (mg/L)	100	Pb (mg/L)	5	Sr (mg/L)	20
SO_4 (mg/L)	192	Li (mg/L)	0.1	Tl (µg/L)	2
NH_3-N (mg/L)	30	Mn (mg/L)	0.2	U (µg/L)	30
V (mg/L)	0.1	Phenol (mg/L)	11	Oil &Grease	10
Zn (mg/L)	2	Total Ra (pCi/L)	5	RSC (meq/L)	1.25
Gross alpha particle radioactivity (including Ra 226 but excluding Radon and Uranium) (pCi/L)					15

What Constituents Should We Analyze? NPDES+ List

- Published in Water 2022, 14(14), 2162; https://doi.org/10.3390/w14142162
- Water quality standards for surface water discharge, land application, irrigation, wildlife and livestock watering, road application, dust control, and groundwater standards
- Developed a multi-tiered analytical approach with a comprehensive analytical list for characterization of physical, chemical, and biological properties of raw produced water and treated produced water using target and non-target analyses as well risks and toxicity assessment



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Review

Analysis of Regulatory Framework for Produced Water Management and Reuse in Major Oil- and Gas-Producing Regions in the United States

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* Correspondence: pxu@nmsu.edu

Abstract: The rapid development of unconventional oil and gas (O&G) extraction around the world produces a significant amount of wastewater that requires appropriate management and disposal. Produced water (PW) is primarily disposed of through saltwater disposal wells, and other reuse/disposal methods include using PW for hydraulic fracturing, enhanced oil recovery, well drilling, evaporation ponds or seepage pits within the O&G field, and transferring PW offsite for management or reuse. Currently, 1–2% of PW in the U.S. is used outside the O&G field after treatment. With the considerable interest in PW reuse to reduce environmental implications and alleviate regional water scarcity, it is imperative to analyze the current regulatory framework for PW management and reuse. In the U.S., PW is subject to a complex set of federal, state, and sometimes local regulations to address the wide range of PW management, construction, and operation practices. Under the supervision of the U.S. Environment Protection Agency (U.S. EPA), different states have their own regulatory agencies and requirements based on state-specific practices and laws. This study analyzed the regulatory framework in major O&G-producing regions surrounding the management of PW, including relevant laws and jurisdictional illustrations of water rules and responsibilities, water quality standards, and PW disposal and current/potential beneficial reuse up to early 2022. The selected eastern states (based on the 98th meridian designated by the U.S. EPA as a tool to separate discharge permitting) include the Appalachian Basin (Marcellus and Utica shale areas of Pennsylvania, Ohio, and West Virginia), Oklahoma, and Texas; and the western states include California, Colorado, New Mexico, and Wyoming. These regions represent different regulations; climates; water quantities; quality diversities; and geologic, geographic, and hydrologic conditions. This review is particularly focused on the water quality standards, reuse practices and scenarios, risks assessment, knowledge gaps, and research needs for the potential reuse of treated PW outside of O&G fields. Given the complexity surrounding PW regulations and rules, this study is intended as preliminary guidance for PW management, and for identifying the knowledge gaps and research needs to reduce the potential impacts of treated PW reuse on the environment and public health. The regulations and experiences learned from these case studies would significantly benefit other states and countries with O&G sources for the protection of their environment and public health.

Keywords: produced water; water reuse; regulatory framework; water quality standards; Appalachian Basin; California; Colorado; New Mexico; Oklahoma; Texas; Wyoming

check for updates

Citation: Jiang, W.; Lin, L.; Xu, X.; Wang, H.; Xu, P. Analysis of Regulatory Framework for Produced Water Management and Reuse in Major Oil- and Gas-Producing Regions in the United States. *Water* **2022**, *14*, 2162. https://doi.org/10.3390/w14142162

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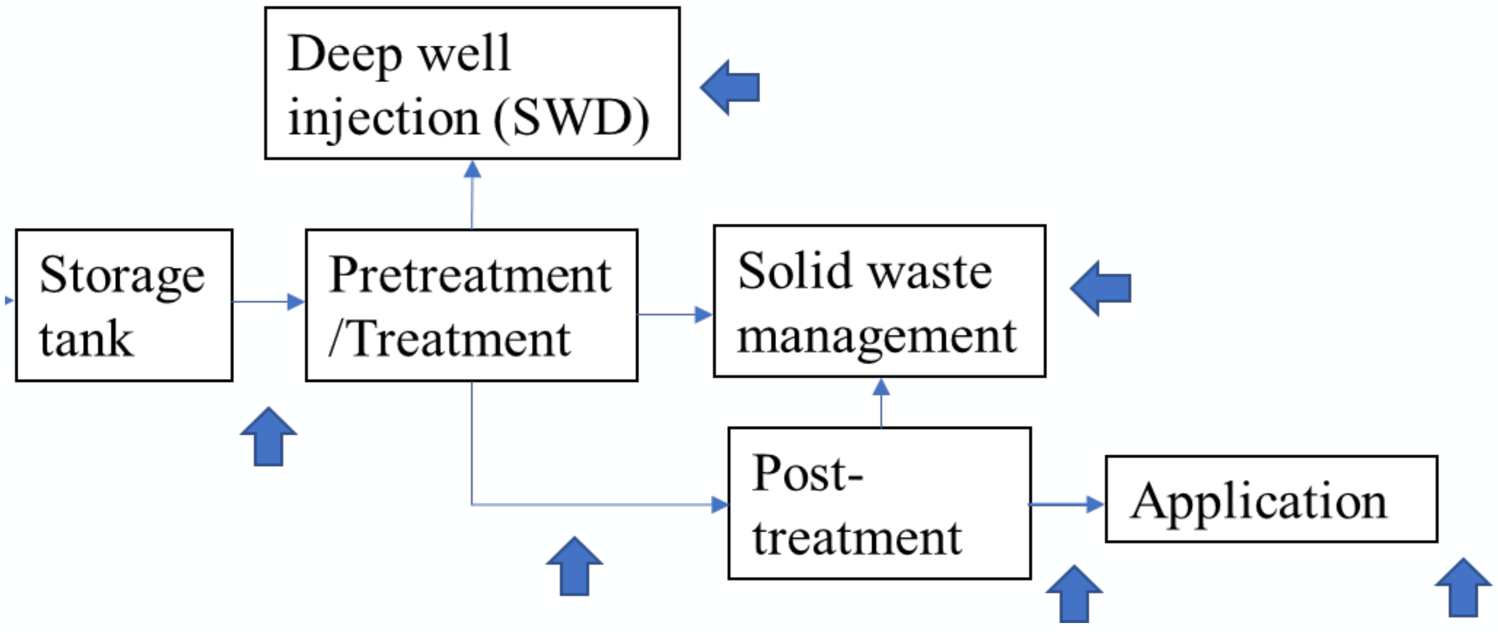
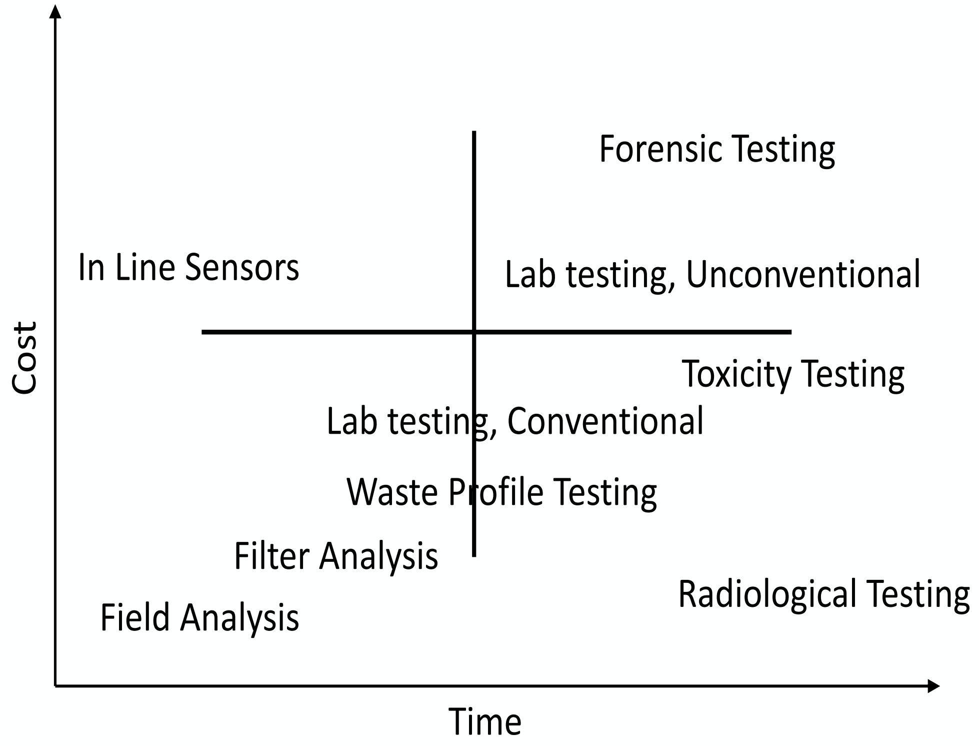
1. Introduction

A significant amount of produced water (PW) is brought to the land surface during oil and gas (O&G) exploration and production [1]. PW is primarily composed of reservoir water extracted from rock formation (i.e., formation water); it may also include a portion of the frac fluid returned to the surface after hydraulic fracturing (i.e., HF flowback water) or the water injected for enhanced oil recovery (EOR). PW contains the naturally occurring

Multi-tiered Approach for Produced Water Characterization





The cost and turnaround time of produced water analysis

Multi-tiered Approach for Produced Water Characterization
NPDES+ List

Level	Use	Parameters	Frequency	Sample
Tier 1	Continuous monitoring, bulk testing, rapid analysis, process control	Flow TSS/Turbidity TDS/EC TOC/DOC/COD pH ORP Iron (total, dissolved, Fe^{2+}) H_2S NH_3 Alkalinity Hardness (total, dissolved) Specific gravity Percent Moisture Optional: UV-Vis, Fluorescence excitation-emission matrix (F-EEM)	Baseline, real-time, continuous, and routine	Feed/produced water Product water Brine

Multi-tiered Approach for Produced Water Characterization
NPDES+ List

Level	Use	Parameters	Frequency	Sample

Tier 2	Detailed characterization, routine monitoring, and Tier 1 data verification	**Inorganics**
		• Metal elements (33), SW-864 6020A, dissolved, total Hg, SW-846 7470
		• Anions (7), EPA 300
		• Radionuclides
		  • Radium 226, 228
		  • Gross Alpha/Beta
		  • U 235, 236, 238
		  • Strontium 90
			Baseline (at least once)
			Demonstrating treatment efficacy and reliability, beneficial reuse investigation
				Feed/produced water
				
				Product water
				
				Brine

Multi-tiered Approach for Produced Water Characterization
NPDES+ List

Level	Use	Parameters	Frequency	Sample

Tier 2	Detailed characterization, routine monitoring, and Tier 1 data verification	**Organics**
		• Oil and Grease
		• GRO [C6-C10] by 8015D
		• DRO [C10-C28] by 8015D
		• MRO (C28-40) by 8015D
		• VOCs SW-846 8260 (91)
		• SVOC - General by 8270E (139)
		• SVOC - TPH by 8015 (8)
		• 1-2 samples for screening:
		  • VOC - TPH by 8015
		  • SVOC - Explosives by 8330B
		  • SVOC - Agent Breakdown Products
		  • SVOC - Pesticides/Herbicides by 8081B
		  • SVOC - Polychlorinated biphenyls (PCBs) (8280A)
		  • SVOC - PAHs
		  • SVOC - Organic Acids by 8015D
		  • SVOC – Dioxins
		  • TOX by SW 846 9020
		  • PFOA, PFOS & PFHxS by EPA 537.1 Modified
			Baseline (at least once), Demonstrating treatment efficacy and reliability, beneficial reuse investigation	Feed/ produced water
				
				Product water
				
				Brine

Multi-tiered Approach for Produced Water Characterization NPDES+ List

Level	Use	Parameters	Frequency
Tier 3	Risks and toxicology assessment Fate/transport modeling.	WET Testing acute and chronic toxicity	Product water (at least once)
		HiRes LC-MS non-target screening	
		Analysis of treated effluent on soil, plant, tissue samples	
Tier 4	Waste and residual characterization	Mass balance	As needed

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Where Do We Analyze Produced Water?

- **Request for Information on Analytical Capabilities**
  - Contacted >20 commercial labs and research labs to develop a database
    - Establish costs and throughput of current analytical methods
    - Identify potential issues and future analytical, toxicology, or method development needs
    - Use information to better quantify Tier sampling and analysis requirements

REQUEST FOR INFORMATION
ANALYTICAL CAPABILITIES

NEW MEXICO PRODUCED WATER RESEARCH CONSORTIUM

JUNE, 2020

Produced Water Sampling Protocol

NM STATE UNIVERSITY

GUIDANCE ON TREATED AND UNTREATED PRODUCED WATER SAMPLING PROCEDURE

NEW MEXICO PRODUCED WATER RESEARCH CONSORTIUM

NOVEMBER 1, 2022

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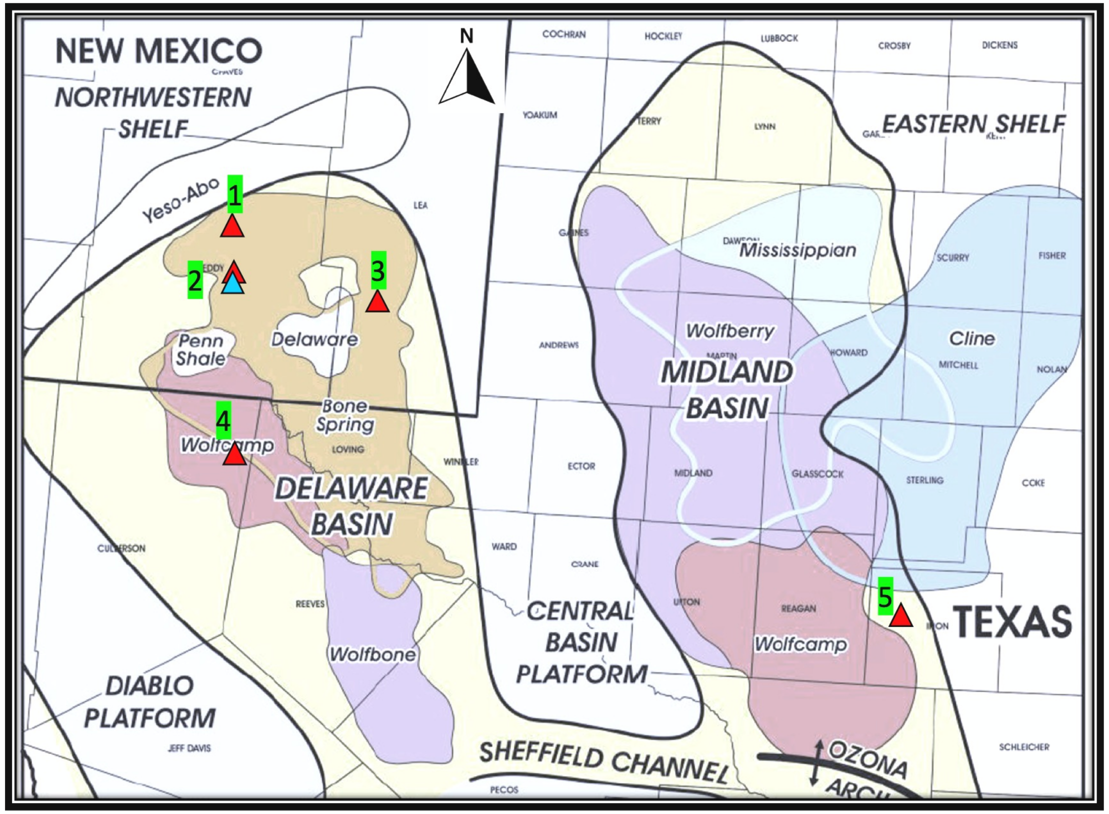
Characterization of Produced Water in the Permian Basin

- Produced water quality is highly variable: by region, within an oil or gas play, with time
- Limited produced water quality data in existing database: primarily inorganic ions

	Permian Basin	Wolfcamp Formation	Delaware Formation	Artesia Formation	Yeso Formation	Bone Spring Formation	San Andres Formation
TDS (mg/L)	10,048-384,963/ 118,253	12,136-249,459/ 95,096	12,708-360,545/ 185,433	10,050-384,963/ 94,584	10,818-381,108/ 123,784	10,048-255,451/ 105,569	10,026-391,007/ 118,879
pH	0.5-11.7/6.8	4.5-8.6/7.0	4.8-8.9/6.9	4.6-9.7/7.1	0.5-8.8/6.7	6.3-7.1/6.8	0.6-11.7/6.9
Mg (mg/L)	3-27,910/ 1,901	84-5,965/ 1,103	3-10,800/ 2,509	12-18,400/ 1,593	12-18,980/ 2,281	54.4-3396.6/ 760	2.7-27,910/ 2,087
Ca (mg/L)	24-60,073/ 6,051	211-40,800/ 6,358	24-46,346/ 12,992	87-25,315/ 3,205	235-40,420/ 6,996	174.5-21,720/ 3347	107-60,073/ 6,952
Cl (mg/L)	40-245,700/ 71,224	3,951-151,900/ 56,362	2,460-225,612/ 113,116	3,794-222,596/ 56,580	2,350-237,245/ 74,606	4,076-156,699/ 60,184	40-245,700/ 70,738
Na (mg/L)	209-143,086/ 71,224	2,625-54,068/ 29,045	5,253-109,024/ 51,113	209-128,175/ 37,470	1,529-107,396/ 35,948	1,982-80,469/ 30,723	1,123-143,086/ 35,479
K (mg/L)	14-33,962/ 861	97-742/ 362	79-1,454/ 548	65-4,620/ 505	14-1,570/ 472	109.8-1,232/ 365	8-33,962/ 1,622
Sulfate (mg/L)	18-12,320/ 2,131	84-12,080/ 1,753	84-6,280/ 1,523	18-11,900/ 2,294	35-11,800/ 2,211	111-5,250/ 1,420	22.4-12,320/ 2,362
Br (mg/L)	10-1,064/ 430	10 - 756/ 390	NA	NA	240-963/ 481	152-1,065/ 382	17-517/ 153
HCO3 (mg/L)	5-7,440/ 731	5-4,204/ 619	5-5,558/ 376	9-7,440/ 878	5-3,851/ 645	5-891/ 390	7-3,960/ 663
TOC (mg/L)	53-184/123	86-184/138	NA	NA	NA	119	NA

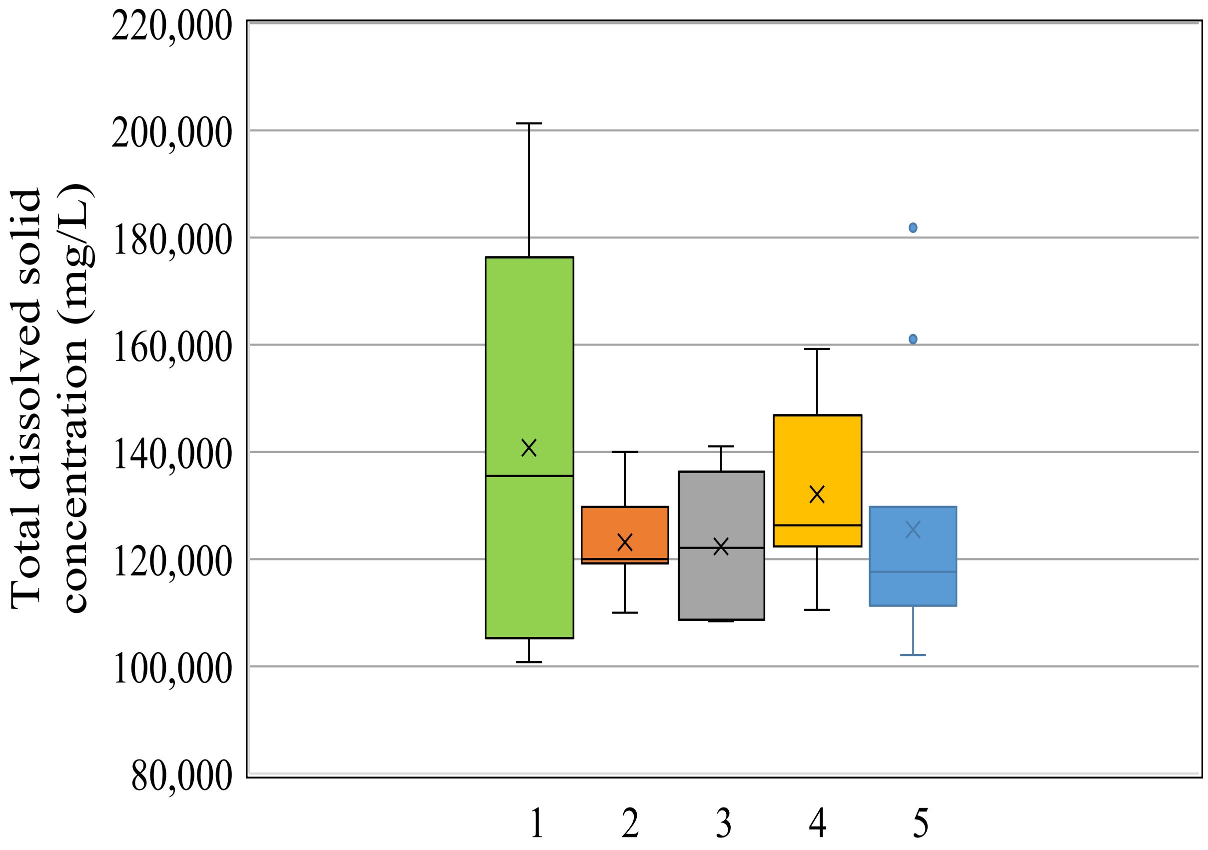
Sampling points of 46 PW and 10 Pecos River water



Produced water sampling point

Pecos River water sampling point

TDS distribution of PW at different sampling points



Source: Jiang et al., JHM 2022, 430, 128409

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Chemical Analysis

More than 300 targeted analytes were quantitatively analyzed, including wet chemistry, inorganics, radionuclides, organics such as VOCs, SVOCs, total petroleum hydrocarbons, organic acids, oil and grease, pesticides/herbicides, dioxins, and tentatively identified compounds, and per- and polyfluoroalkyl substances (PFAS).

For 10 produced water samples collected in 2020, 91 analytes were quantified and 218 analytes were not detected (309 in total)

For 10 Pecos River samples collected in 2020, 67 analytes were quantified and 242 analytes were not detected (309 in total)

Source: Jiang et al., JHM 2022, 430, 128409

Water Quality Characterization

Statistical results of general quality parameters of the 46 PW samples collected from the Delaware and Midland Basins

		Mean	Max	Min	25th percentile	50th percentile	75th percentile
Alkalinity	mg/L as $CaCO_3$	272	870	100	128	207	336
Ammonia	mg/L	432	750	320	330	400	495
COD	mg/L	1,626	3,100	930	1,250	1,400	1,950
pH	SU	6.6	8.1	3.9	6.3	6.7	7.0
TDS	mg/L	128,641	201,474	100,830	113,441	122,280	134,525
TOC	mg/L	103.5	248.1	2.4	28	90.6	173.3
TSS	mg/L	342.9	790	85	142.5	375	422.5
Turbidity	NTU	116.4	200	23	36	110	200
MBAS	mg/L	1.10	2.1	0.047	0.92	0.97	1.33

Source: Jiang et al., JHM 2022, 430, 128409

Water Quality Characterization

Produced Water		Average	Max	Min
Radionuclide				
Gross Alpha	pCi/L	1105.6	1630	660
Gross Beta	pCi/L	874.6	1230	456
Radium-226	pCi/L	43.92	111	0.736
Radium-228	pCi/L	151.27	291	2.56

Water quality of Pecos River water samples		Average	Max	Min	Drinking water standards
Radionuclide					
Gross Alpha	**pCi/L**	**24.6**	**39.8**	**7.7**	**15**
Gross Beta	pCi/L	14.1	24.2	1.4	4 millirems per year
Radium-226	pCi/L	3.56	29.9	0.1	5 pCi/L for
Radium-228	pCi/L	0.42	0.8	0.2	Combined Ra226/228

Source: Jiang et al., JHM 2022, 430, 128409

Water Quality Characterization

Produced Water **VOCs**		Average	Max	Min
Benzene	mg/L	2.61	4.90	1.90
Ethylbenzene	mg/L	0.11	0.16	0.07
Toluene	mg/L	2.53	3.70	1.70
Xylenes, Total	mg/L	1.19	1.60	0.71

No VOCs detected in Pecos River (9 samples)

Source: Jiang et al., JHM 2022, 430, 128409

Water Quality Characterization

Produced Water Samples		Average	Max	Min
Oil and Others				
Diesel Range Organics (C10-C20)	ug/L	45,750	130,000	22,000
Gasoline Range Organics [C6 - C10]	ug/L	21,625	46,000	13,000
Motor oil/lube range organics (MRO) (C20-C34)	ug/L	32,444	97,000	12,000
Tributyl phosphate	ug/L	34.6	74	3.3
Tentatively Identified Compound	ug/L	531	1000	280

Pecos River water samples		Average	Max	Min
Oil and Others				
Gasoline Range Organics [C6 - C10]	ug/L		54	ND
Motor oil/lube range organics (MRO) (C20-C34)	ug/L	230	310	180
Tributyl phosphate	ug/L	3.6	5.7	1.7
Tentatively Identified Compound	ug/L	-	55	-

Source: Jiang et al., JHM 2022, 430, 128409

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Water Quality Characterization

Produced water		Average	Max	Min
Organic - SVOC - General		Average	Max	min
1,1'-Biphenyl	ug/L	5.9	8.5	3.8
1,4-Dioxane	ug/L		21	ND
1-Methylnaphthalene	ug/L	23	36	15
2-Methylnaphthalene	ug/L	38	65	26
2-Methylphenol	ug/L	82	98	68
2,4-Dimethylphenol	ug/L	34	42	29
Ethylene glycol	mg/L		27	ND
Methylphenol, 3 & 4	ug/L	90	110	72
Phenol	ug/L	203	250	170
Pyridine	ug/L	238	300	120

Not detected in Pecos River (9 samples)

Source: Jiang et al., JHM 2022, 430, 128409

Water Quality Characterization

Produced Water		Average	Max	Min
Organic - SVOC - Pesticides/Herbicides				
alpha-BHC	ug/L	0.018	0.027	0.0088
Endosulfan I	ug/L	0.855	0.98	0.73
Endrin	ug/L		0.0038	ND

Pecos River water		**Average**	**Max**	**Min**
Organic - SVOC - Pesticides/Herbicides				
Endosulfan I	ug/L	0.00405	0.0042	0.0039
4,4'-DDD	ug/L		0.01	ND
4,4'-DDT	ug/L		0.0057	ND

Source: Jiang et al., JHM 2022, 430, 128409

Water Quality Characterization

Produced Water		Average	Max	Min
Organic - SVOC - PAH				
Anthracene	ug/L		1.1	ND
Naphthalene	ug/L	15.44	24	11
Phenanthrene	ug/L	3.76	6.6	2.7
Fluorene	ug/L	4.35	5.6	3.1

Pecos River water		**Average**	**Max**	**Min**
Organic - SVOC - PAH				
Naphthalene	ug/L		6	ND
Fluorene	ug/L		1.2	ND

Source: Jiang et al., JHM 2022, 430, 128409

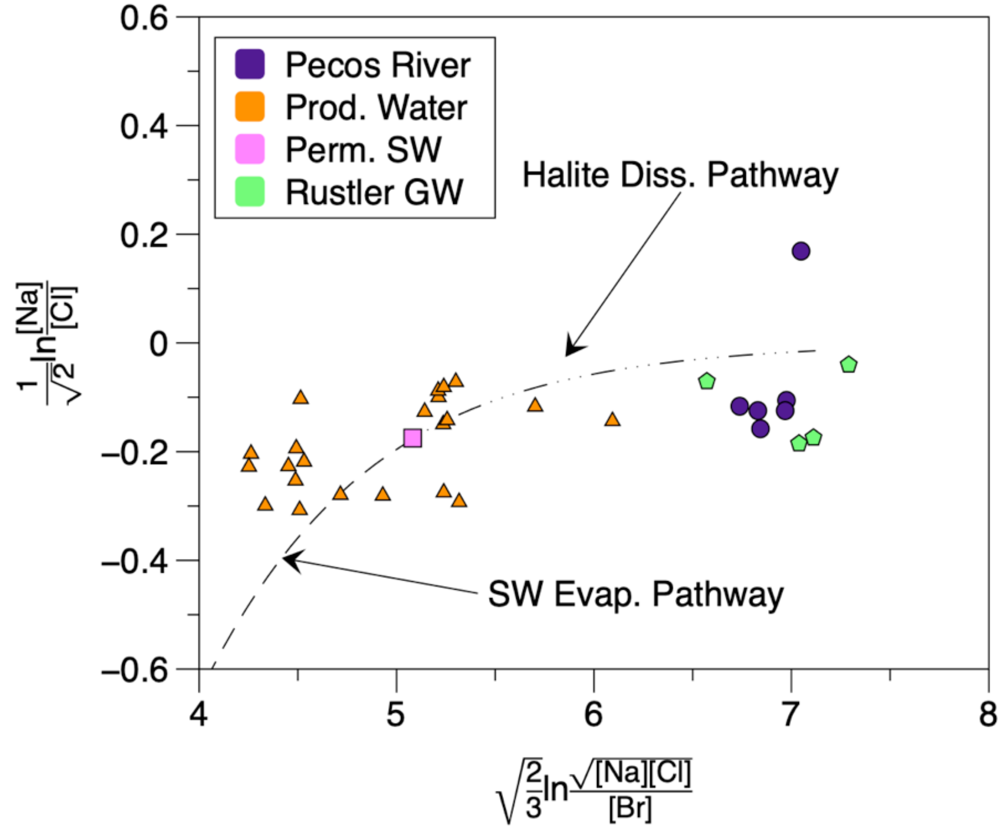
Perfluoroalkyl substances (PFAS)		Produced Water	Pecos River
Perfluorobutanesulfonic acid (PFBS)	ng/L	0.17	2
Perfluorobutanoic acid (PFBA)	ng/L	0.31	1.3
Perfluorodecanesulfonic acid (PFDS)	ng/L	ND	ND
Perfluorodecanoic acid (PFDA)	ng/L	ND	ND
Perfluorododecanesulfonic acid (PFDoS)	ng/L	ND	ND
Perfluorododecanoic acid (PFDoA)	ng/L	ND	ND
Perfluoroheptanesulfonic Acid (PFHpS)	ng/L	ND	ND
Perfluoroheptanoic acid (PFHpA)	ng/L	ND	0.35
Perfluorohexanesulfonic acid (PFHxS)	ng/L	0.25	1
Perfluorohexanoic acid (PFHxA)	ng/L	ND	1.2
Perfluorononanesulfonic acid (PFNS)	ng/L	ND	ND
Perfluorononanoic acid (PFNA)	ng/L	ND	ND
Perfluorooctanesulfonamide (FOSA)	ng/L	ND	0.54
Perfluorooctanesulfonic acid (PFOS)	ng/L	ND	1.2
Perfluorooctanoic acid (PFOA)	ng/L	ND	1
Perfluoropentanesulfonic acid (PFPeS)	ng/L	ND	0.24
Perfluoropentanoic acid (PFPeA)	ng/L	ND	1.8
Perfluorotetradecanoic acid (PFTeA)	ng/L	0.24	ND
Perfluorotridecanoic acid (PFTriA)	ng/L	ND	ND
Perfluoroundecanoic acid (PFUnA)	ng/L	ND	ND

Preliminary PFAS Results of 1 Produced Water Sample (5/34 detected) and 1 Pecos River Sample (10/34 compounds detected)

Based on FracFocus database, no PFAS were used in HF chemical additives in the Permian Basin.

Source: Jiang et al., JHM 2022, 430, 128409

Water Quality Characterization



TDS of Pecos River water varies between 2290 – 6200 (average 4420) mg/L

Strongly overlapping data of these conservative relationships suggest that shallow brines from evaporite mineral dissolution is the dominant source of salinity to the Pecos River samples.

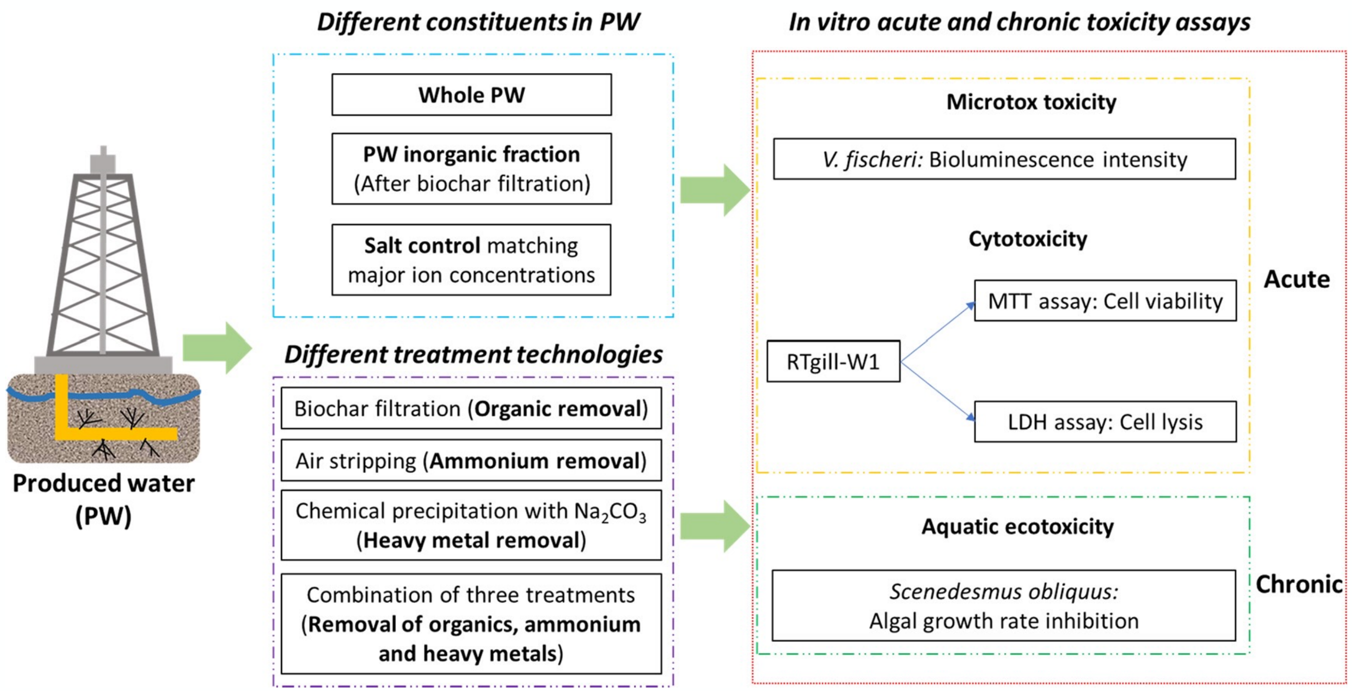
Isometric log-ratio Na-Cl-Br plot showing data from Permian Basin PW, Pecos River, and Rustler aquifer groundwater samples against modeled pathways for ancient (late Permian) seawater evaporation and halite mineral dissolution.

Jiang et al., JHM 2022, 430, 128409

Toxicological Characterization of Produced Water from the Permian Basin

- PW toxicity was studied using in vitro toxicity assays using various aquatic organisms (luminescent bacterium , fish gill cell line RTgill-W1, and microalgae).

- High salinity was the foremost toxicological driver in PW, followed by organic contaminants.
- Treatment required to reduce toxicity:
    - Salts - Desalination
    - Organic removal
    - Ammonia removal
    - Heavy metals removal



Source: Hu et al., Sci. Total Environ 2022, 815, 152943

Journal of Hazardous Materials 430 (2022) 128409

Contents lists available at ScienceDirect

Journal of Hazardous Materials

journal homepage: www.elsevier.com/locate/jhazmat

Research Paper

Characterization of produced water and surrounding surface water in the Permian Basin, the United States

Wenbin Jiang[a], Xuesong Xu[a], Ryan Hall[b], Yanyan Zhang[a], Kenneth C. Carroll[c], Frank Ramos[d], Mark A. Engle[e], Lu Lin[a], Huiyao Wang[a], Matthias Sayer[b], Pei Xu[a,*]

Data in Brief 43 (2022) 108443

Contents lists available at ScienceDirect

Data in Brief

journal homepage: www.elsevier.com/locate/dib

Data Article

Datasets associated with the characterization of produced water and Pecos River water in the Permian Basin, the United States

Wenbin Jiang[a], Xuesong Xu[a], Ryan Hall[b], Yanyan Zhang[a], Kenneth C. Carroll[c], Frank Ramos[d], Mark A. Engle[e], Lu Lin[a], Huiyao Wang[a], Matthias Sayer[b], Pei Xu[a,*]

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Science of the Total Environment 815 (2022) 152943

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Science of the Total Environment

journal homepage: www.elsevier.com/locate/scitotenv

Toxicological characterization of produced water from the Permian Basin

Lei Hu[a], Wenbin Jiang[a], Xuesong Xu[a], Huiyao Wang[a], Kenneth C. Carroll[b], Pei Xu[a], Yanyan Zhang[a,*]

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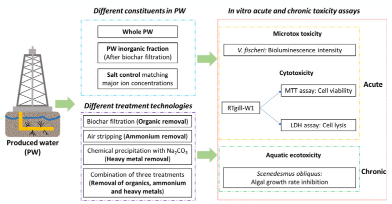
[b] Department of Plant and Environmental Science, New Mexico State University, Las Cruces, NM 88003, USA

HIGHLIGHTS

- High salinity was the predominant toxicological driver in PW.
- Organic contaminants had a significant impact on PW toxicity.
- Heavy metals and ammonium in PW also contribute to toxicity.
- Toxicity assays had different sensitives to the chemical constituents present in PW.

GRAPHICAL ABSTRACT



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ABSTRACT

Produced water (PW) is a hypersaline waste stream generated from the shale oil and gas industry, consisting of numerous anthropogenic and geogenic compounds. Despite prior geochemical characterization, the comprehensive toxicity assessment is lacking for evaluating treatment technologies and the beneficial use of PW. In this study, a suite of *in vitro* toxicity assays using various aquatic organisms (luminescent bacterium *Vibrio fischeri*, fish gill cell line RTgill-W1, and microalgae *Scenedesmus obliquus*) were developed to investigate the toxicological characterizations of PW from the Permian Basin. The exposure to PW, PW inorganic fraction (PW-IF), and PW salt control (PW-SC) at 30 – 50% dilutions caused significant toxicological effects in all model species, revealing the high salinity was the foremost toxicological driver in PW. In addition, the toxicity level of PW was usually higher than that of PW-IF, suggesting that organic contaminants might also play a critical role in PW toxicity. When comparing the observed toxicity with associated chemical characterizations in different PW samples, strong correlations were found between them since higher concentrations of contaminants could generally result in higher toxicity towards exposed organisms. Furthermore, the toxicity results from the pretreated PW indicated that those *in vitro* toxicity assays had different sensitives to the chemical components present in PW. As expected, the combination of multiple pretreatments could lead to a more significant decrease in toxicity compared to the single pretreatment since the mixture of contaminants in PW might exhibit synergistic toxicity. Overall, the current work is expected to enhance our understanding of the potential toxicological impacts of PW to aquatic ecosystems and the relationships between the chemical profiles and observed toxicity in PW, which might be conducive to the establishment of monitoring, remediation, treatment, and reuse protocols for PW.

1. Introduction

According to the U.S. Energy Information Administration (EIA, 2015; Kuuskraa et al., 2013), approximately 55 billion m^3 of shale oil and 207 trillion m^3 of shale gas are technically recoverable globally, which can satisfy the world's energy supply for over 100 years. The rapid expansion of horizontal drilling and hydraulic fracturing practices has improved the oil and gas production from shale formations, thereby promoting the United

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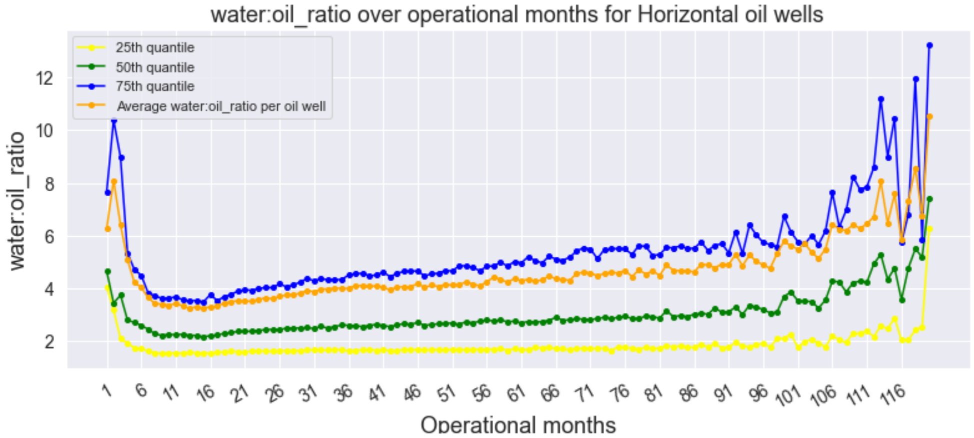
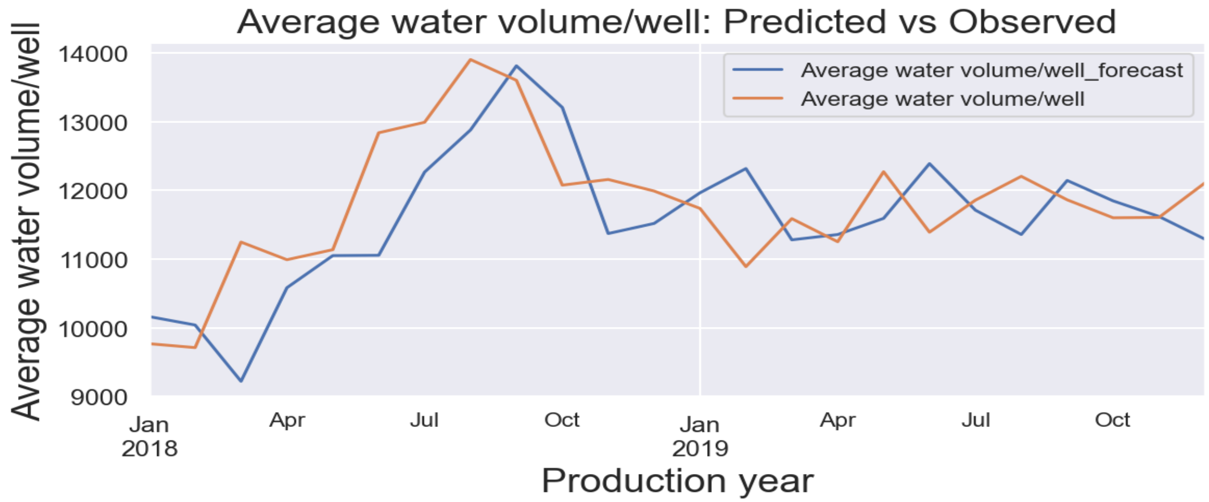
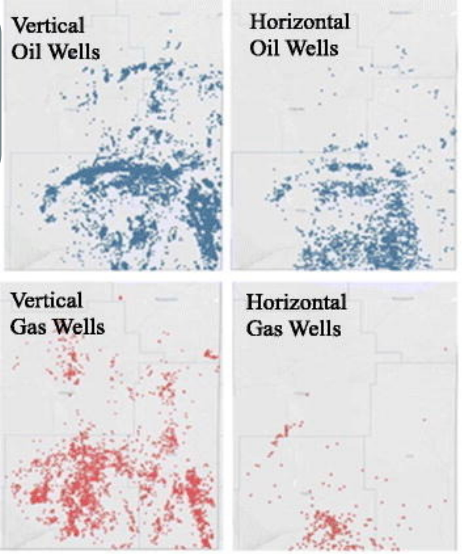
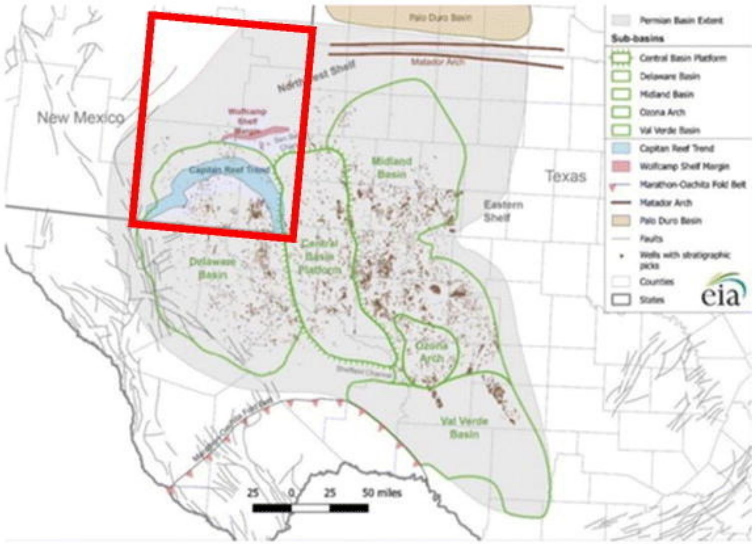
Analysis and prediction of produced water quantity in the Permian Basin using machine learning techniques

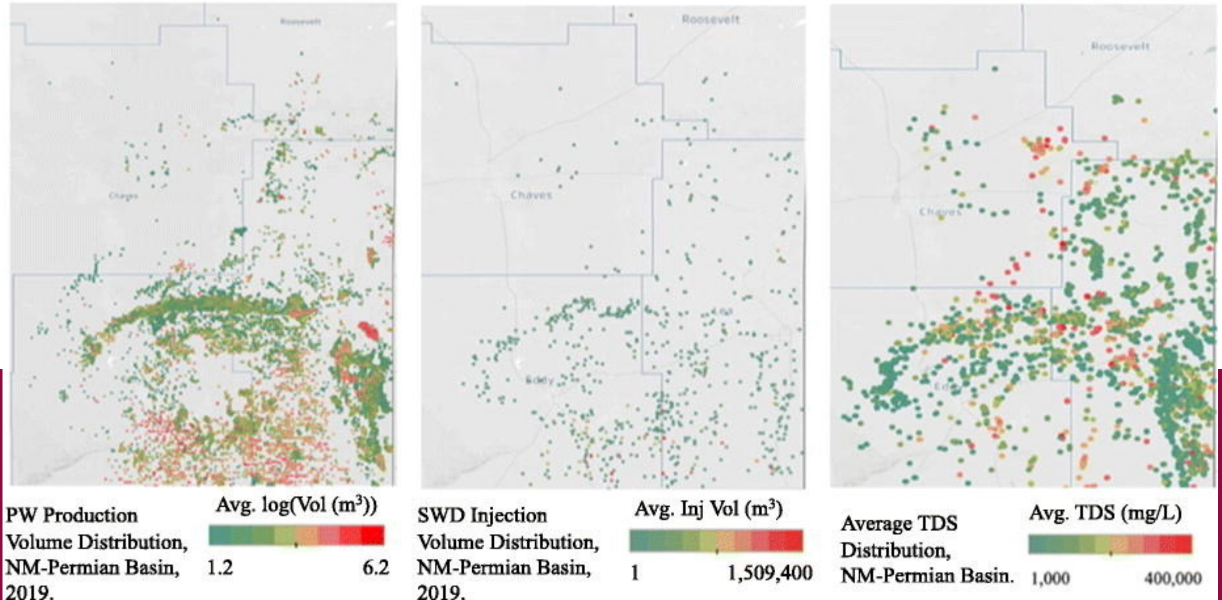
Data Collection (NMOCD FTP Server) → Data Pre-processing → Initial Data Analysis → Regression Analysis → Statistical Analysis











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Analysis and prediction of produced water quantity and quality in the Permian Basin using machine learning techniques

Wenbin Jiang [a], Beepana Pokharel [b], Lu Lin [a], Huiping Cao [b], Kenneth C. Carroll [c], Yanyan Zhang [a], Carlos Galdeano [d], Deepak A. Musale [d], Ganesh L. Ghurye [d], Pei Xu [a,*]

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[d] *ExxonMobil Upstream Research Company, Research & Technology Development-Unconventionals, Spring, TX 77389, United States*

HIGHLIGHTS

GRAPHICAL ABSTRACT

Ongoing and Collaborative Produced Water Characterization Studies

- Characterization of San Juan Basin produced water treated via sea water reverse osmosis (SWRO) - Himali Delanka-Pedige, NMSU
- Non-target analysis of produced water and treated PW - Robert Young, NMSU
- Toxicity analysis of thermal-desalinated water, SWRO permeate, and posttreatment to reduce toxicity – Yanyan Zhang, NMSU
- Collaboration with EPA on characterization of produced water samples from the Permian Basin and San Juan Basin - Sean Thimons, ORISE – EPA
- Treatment of Permian Basin produced water and water quality characterization – James Rosenblum, CSM; and Josh Butler, ExxonMobil

Highlights of Accomplishments

- Developed foundational documents such as produced water research roadmaps and gaps in analysis, research plan, testing guidance, and sampling protocols
- Reviewed current regulatory framework on produced water production, management and reuse including water quality standards in nine oil and gas producing states, including CA, CO, NM, OH, OK, PA, TX, WV, WY.
- Developed a multi-tiered analytical approach with a comprehensive analytical list (NPDES+) for characterization of physical, chemical, and biological properties of raw produced water and treated produced water using target and non-target analyses as well risks and toxicity assessment
- Over 300 targeted analytes were quantitatively analyzed in PW samples and the Pecos River. Provides baseline analytical information to advance PW research for potential reuse and fills the knowledge gap regarding PW quality to support science-based decision making.

Highlights of Accomplishments

- Developed state-of-the-science in produced water quality analysis methods
- Improved characterization of physical, chemical, microbiological, and environmental toxicity analysis of produced water and treated produced water from San Juan and Permian Basins
- Evaluated treatment efficiency of desalination (reverse osmosis, and thermal distillation) and post-treatment
- Statistically characterized produced water quality and quantity in the Permian Basin

2023/24 Goals and Objectives

- Conduct bench- and pilot-scale testing, characterize the quality of PW and treated water, including target analysis, toxicity study, and non-target analysis of “unknown” constituents.
- Better understand the transport and fate of constituents in PW during treatment and beneficial use applications.
- Develop a transparent, environmental and human health risk assessment framework for the beneficial reuse of treated PW that is protective of human health and the environment.
- Provide science and knowledge to assist in regulations, determining management strategies, selecting treatment methods, evaluating risks and environmental impacts specific to intended beneficial use of treated PW.

NMSU Research Publications

1. Jiang, W., Lin, L., Xu, X., Wang, H., Xu, P. (2022) Analysis of regulatory framework for produced water management and reuse in major oil and gas producing regions in the United States. Water 14 (14), 2162. https://www.mdpi.com/2073-4441/14/14/2162

"This study reviews the current regulatory framework for produced water production, management, and reuse in the major oil and gas production areas in the U.S., including Appalachian Basin, California, Colorado, New Mexico, Oklahoma, Texas, and Wyoming."

2. Sabie, R.P., Pillsbury, L., and Xu, P. (2022). Spatiotemporal Analysis of Produced Water Demand for Fit-For-Purpose Reuse—A Permian Basin, New Mexico Case Study. Water 14 (11), 1735. https://www.mdpi.com/2073-4441/14/11/1735

"In this study, a generalized framework was developed for estimating produced water (PW) supply and potential demand for treated PW reuse in agriculture, dust suppression, power generation, and river flow augmentation using Eddy and Lea counties, New Mexico as a case study"

3. Tidwell, V., Gunda, T., Caballero, M., Xu, P., Xu, X., Bernknopf, R., Broadbent, C., Malczynski, L.A., Jacobson, J. (2022) Produced Water-Economic, Socio, Environmental Simulation Model (PW-ESEim) Model: Proof-of-Concept for Southeastern New Mexico. SAND2022-6636R. Published by Sandia National Lab.(SNL-NM), Albuquerque, NM (United States). https://www.osti.gov/servlets/purl/1868149

"A proof-of-concept tool, the Produced Water-Economic, Socio, Environmental Simulation model (PW-ESESim), was developed to support ease of analysis. The tool was designed to facilitate head-to -head comparison of alternative produced water sources, treatment, and reuse water management strategies. A graphical user interface (GUI) guides the user through the selection and design of alternative produced water treatment and reuse strategies and the associated health and safety risk and economic benefits."

4. Jiang, W., Xu, X., Hall, R., Zhang, Y., Carroll, K.C., Ramos, F., Engle, M.A., Lin, L., Wang, H., Sayer, M., Xu, P. (2022). Characterization of Produced Water and Surrounding Surface Water in the Permian Basin, the United States. Journal of Hazardous Materials. 430, 128409. https://doi.org/10.1016/j.jhazmat.2022.128409

"In this research, over 300 analytes for organics, inorganics, and radionuclides were quantitatively analyzed in produced water (PW) samples from the Permian Basin and in surface water samples from the Pecos River in New Mexico. This study provides baseline analytical information to advance PW research for potential reuse and fills the knowledge gap regarding PW quality to support science-based decision making.

5. Jiang, W., Xu, X., Hall, R., Zhang, Y., Carroll, K.C., Ramos, F., Engle, M.A., Lin, L., Wang, H., Sayer, M., Xu, P. (2022). Datasets associated with the characterization of produced water and Pecos River water in the Permian Basin, the United States. Data in Brief, 43, 108443. https://www.sciencedirect.com/science/article/pii/S2352340922006400

"This paper presents data related to the analysis of produced water and river water samples in the Permian Basin with a specific focus on wet chemistry, mineral salts, metals, oil and grease, volatile and semi-volatile organic compounds, radionuclides, ammonia, hydraulic fracturing additives, and per- and polyfluoroalkyl substances."

NMSU Research Publications

6. Hu, L., Jiang, W., Xu, X., Wang, H., Carroll, K.C., Xu, P., Zhang, Y. (2022). Toxicological characterization of produced water from the Permian Basin. Science of The Total Environment. 815(1), 152943. https://doi.org/10.1016/j.scitotenv.2022.152943

"In this study, an in vitro toxicity assessment was conducted using aquatic microorganisms to explore toxicological characteristics of produced water (PW) from the Permian Basin, New Mexico. It was found that high salinity, organic contaminants, metals, and ammonia present in PW are major toxicity drivers and need to be removed for fit-for-purpose beneficial uses of treated PW "

7. Thakur, P., Ward, A.L., Schaub, T.M. (2022). Occurrence and behavior of uranium and thorium series radionuclides in the Permian shale hydraulic fracturing wastes. Environmental Science and Pollution Research 29 (28), 43058-43071. https://link.springer.com/article/10.1007/s11356-021-18022-z

"This study explored the risk of releasing radioactive materials during the oil and gas recovery process in the Permian Basin, New Mexico. The results confirmed the presence of radioactive materials (^{224}Ra, ^{226}Ra, ^{228}Ra) in addition to dissolved salts, divalent cations, and high total dissolved solids in the hydraulic fracturing wastes."

8. Chen, L., Wang, H., Xu, P. (2022). Photocatalytic membrane reactors for produced water treatment and reuse: fundamentals, affecting factors, rational design, and evaluation metrics. Journal of Hazardous Materials, 127493. https://www.sciencedirect.com/science/article/abs/pii/S0304389421024614

"In this study, the potential of photocatalytic membrane reactors (PMR) to treat produced water (PW) was evaluated. The mechanisms of photocatalysis and membrane processes in a PMR, factors affecting PMR performance, rational design, and evaluation metrics for PW treatment were critically reviewed."

9. Jiang, W., Pokharel, B., Lin, L., Cao, H., Carroll, K.C., Zhang, Y., Galdeano, C., Musale, D.A., Ghurye, G.L., Xu, P. (2021). Analysis and Prediction of Produced Water Quantity and Quality in the Permian Basin using Machine Learning Techniques. Science of the Total Environment. 141693. https://www.sciencedirect.com/science/article/abs/pii/S0048969721047689

"In this research, historical produced water (PW) quantity and quality data in the New Mexico portion (NM) of the Permian Basin were comprehensively analyzed, and then, various machine learning algorithms were applied to predict PW quantity for different types of oil and gas wells."

10. Jiang, W., Lin, L., Xu. X., Cheng, X., Zhang, Y., Hall, R., Xu, P. (2021). A Critical Review of Analytical Methods for Comprehensive Characterization of Produced Water. Water, 2021, 13(2), 183; https://doi.org/10.3390/w13020183

"This paper broadly discusses current analytical techniques for produced water characterization, including both standard and research methods. Multi-tiered analytical procedures are proposed including field sampling; sample preservation; pretreatment techniques; basic water quality measurements; organic, inorganic, and radioactive materials analysis; and biological characterization."

NMSU Research Publications

11. Chen, L., Xu, P., Kota, K., Kuravi, S., Wang, H. (2021). Solar distillation of highly saline produced water using low-cost and high-performance carbon black and airlaid paper-based evaporator (CAPER). Chemosphere, 269, 129372.https://doi.org/10.1016/j.chemosphere.2020.129372

"This research introduces a solar-driven carbon black and airlaid paper-based evaporator (CAPER) for desalination of produced water in the Permian Basin, New Mexico. CAPER is low cost, robust, and has the capability of achieving higher removals of salts, heavy metals, Ca, Na, Mg, Mn, Ni, Se, Sr, and V."

12. Hu, L., Wang, H., Xu, P. and Zhang, Y. (2021) Biomineralization of hypersaline produced water using microbially induced calcite precipitation. Water Research, 190, 116753. https://doi.org/10.1016/j.watres.2020.116753

"This study demonstrates the ability of the microbially induced calcite precipitation (MICP) technique that utilizes ureolytic bacteria, to remove Ca^{2+} and toxic contaminants from high salinity produce water for the first time."

13. Chen, L., Xu, P., Wang, H. (2020) Interplay of the Factors Affecting Water Flux and Salt Rejection in Membrane Distillation: A State-of-the-Art Critical Review. Water 2020, 12(10), 2841; https://doi.org/10.3390/w12102841

"This review paper deeply examines the effects of membrane characteristics, feed solution composition, and operating conditions on water flux, mass transport, heat transfer and salt rejection in membrane distillation process."

14. Lu Lin, Wenbing Jiang, Lin Chen, Pei Xu and Huiyao Wang (2020). Treatment of Produced Water with Photocatalysis: Recent Advances, Affecting Factors and Future Research Prospects. Catalysts, 10(8), 924. https://doi.org/10.3390/catal10080924

"This review paper investigated the applicability of photocatalysis-based treatment for produced water (PW) treatment. Factors affecting decontamination, strategies to improve photocatalysis efficiency, recent developments, and future research prospects on photocatalysis-derived systems for PW treatment are discussed here in detail."

15. Alfredo Zendejas Rodriguez, Huiyao Wang, Lei Hu, Yanyan Zhang, and Pei Xu. (2020). Treatment of Produced Water in the Permian Basin for Hydraulic Fracturing: Comparison of Different Coagulation Processes and Innovative Filter Media. Water, 12(3), 770. https://doi.org/10.3390/w12030770

"In this research, chemical coagulation [using $FeCl_3$ and $Al_2(SO_4)_3$] was compared with electrocoagulation (using aluminum electrodes) for their suitability in removing suspended contaminants from produced water for reuse in hydraulic fracturing. The feasibility of several filter media was also studied for refining effluent of the coagulation"

NMSU Research Publications

16. Scanlon, B.R., Reedy, R.C., Xu, P., Engle, M., Nicot, J.P., Yang, Q., and Ikonnikova, S. (2020). Datasets associated with investigating the potential for beneficial reuse of produced water from oil and gas extraction outside of the energy sector. Data in Brief, 105406. https://www.sciencedirect.com/science/article/pii/S2352340920303000

"This article presents data related to volumes of water co-produced with oil and gas production, county-level estimates of annual water use volumes by various sectors, including hydraulic fracturing water use, and the quality of produced water.

17. Scanlon, B.R., Reedy, R.C., Xu, P., Engle, M., Nicot, J.P., Yang, Q., and Ikonnikova, S. (2020). Can we Beneficially Reuse Produced Water from Oil and Gas Extraction in the U.S.? Science of the Total Environment, 717, 137085. https://www.sciencedirect.com/science/article/pii/S0048969720305957

"This study investigated the quantity and the quality of produced water volumes in major U.S. shale oil and gas plays relative to treatment and potential reuse options in irrigation, municipal use, industrial use, surface water and groundwater recharge, and hydraulic fracturing."

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"This study presents an integrated decision support tool to assist in selecting treatment technologies and potential water reuse options for produced water considering the Marcellus Shale in Pennsylvania and the Barnett Shale in Texas as case studies."

Thank YOU!

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