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Review

Hypersaline Wastewater Produced from Pickled Mustard Tuber (Chinese Zhacai): Current Treatment Status and Prospects

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Abstract: Pickled mustard tuber, a worldwide condiment, is increasing at a fast growth rate. Its production generates a considerable amount of hypersaline wastewater containing NaCl of 7 wt.%, COD of 30,000 mg L^{-1}, NH_3-N of 400 mg L^{-1}, and TP of 300 mg L^{-1}. Pickled mustard tuber wastewater (PMTW) has severe effects on crops, deterioration of water quality, soil infertility and ecological systems. Due to the technic difficulties and insufficient support from the local governments; however, PMTW has not yet been widely investigated and well summarized. Therefore, this manuscript reviewed the relatively latest advances in PMTW. Physicochemical and biological hybrid processes mainly treat PMTW and the corresponding cost is 6.00 US dollars per ton. In the context of double carbon capture capacity in China and the development of the pickled mustard industry, PMTW sauce and sustainable reuse such as nutrient recovery, acid and alkaline regeneration and renewable energy may be bright prospects.

Keywords: hypersaline wastewater; pickled mustard tuber; characteristics and environmental effects; treatment arts; prospects arts; resource and energy recovery

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1. Introduction

Food pickling, one of the oldest ways of food preservation, refers to vegetables or fruits soaked in a solution of salt, vinegar, other flavorings and stored for a period, during which the ingredients go through the fermentation process and acquire the desired flavor with naturally-occurring bacteria [1]. There are dozens of pickled recipes, with French cornichons, German sauerkraut and Chinese Zhacai (pickled mustard tuber) being the most well-known and popular three [2]. Pickled mustard tuber plays an important role in Chinese condiments and its consumption is sharply increasing. According to statistical data from (the National Bureau of Statistics), pickled mustard tuber products' consumption increased with an annual growth rate of 6.9% and created a total turnover of USD 0.9 billion in the past five years.

However, the development of mustard tuber products raises some environmental challenges as the amount of hypersaline wastewater is generated during fresh mustard tuber processing. According to our survey results, one-ton of pickled mustard tuber approximately produces 18.5 tons of hypersaline wastewater, containing about 3% NaCl (the maximum NaCl reached 13 wt.%), 7000 mg L^{-1} COD, 400 mg L^{-1} of ammonia-nitrogen and 50 mg L^{-1} phosphorus [3]. Such a high concentration of contaminants, mainly referring to organic matter, ammonia nitrogen, phosphorus nutrients and high salt content, has caused environmental issues like the eutrophication of local rivers, serious soil salinization, and the death of aquatic creatures [4]. These adverse effects raised from the discharge of pickled mustard tuber wastewater (PMTW) have become more evident in recent years. To facilitate this, both Ministry of Ecology and Environment, PRC China and the Bureau of local Ecology and Environment, Chongqing, Sichuan, and Zhejiang, have proposed action plans to prevent and treat hypersaline wastewater pollution of pickled mustard tuber [5]. These plans, on the one hand, regulate the discharge standard of hypersaline pollutants, and on the other hand, they push the development of advanced technologies for hypersaline wastewater treatment.

Based on the number of publications on hypersaline wastewater treatment, no more than 10% of papers focus on PMTW treatment. Given the limited references we can obtain, we summarized the most frequently used techniques of hypersaline wastewater treatment. Hypersaline wastewater treatment mainly involves biological oxidation and physicochemical separation [6]. Biological oxidation is the first choice for high-salt wastewater treatment due to the low cost, excellent resistance to adverse influences, large processing capacity, good treatment effectiveness, and ease of practical implementation [7]. Physicochemical separation is also implemented but used more frequently for higher discharge standard requirements compared to biological oxidation [8,9]. Since biological oxidation is limited by salt inhibition for microorganisms and physicochemical separation is impacted by membrane pollution, neither individual biological oxidation nor physicochemical separation achieves the ideal removal efficiency of pollutants [10]. Thus, the hybrid process of biological oxidation and physicochemical separation is usually selected.

In the context of double carbon capture capacity in China, sustainable energy and resource recoveries are two popular research hotspots and research scopes [11,12]. Theoretically, pickled mustard tuber wastewater has great potential as it is rich in organics, nutrients and salts, according to the research articles and our results. However, PMTW is an environmental issue for Chongqing, Sichuan, Zhejiang, etc., which has not be paid much attention. Technically, the concentrated COD, NH_3-N, TP and salts, cannot be treated by the usual biological or physicochemical processes. Moreover, the high treatment cost of mustard tuber wastewater, and the low profitability of mustard tuber products, results in the enterprises' unwillingness to expend much money treating mustard tuber wastewater. Thirdly, more support from the local governments is required. Therefore, it can be seen that the problems of mustard tuber wastewater treatment do not only include technical difficulties, but also include issues surrounding support from the local governments. Few review papers have reported the latest advances in pickled mustard tuber wastewater treatment to date. This present review thus carries out a short summary of the properties, treatment arts and prospects of PMTW to attract more scholars' attention to hypersaline wastewater produced by the pickled vegetable industry in China and to help figure out sustainable approaches to realize the sustainable reuse of pickled hypersaline wastewater.

2. Pickled Mustard Tuber Wastewater's Characteristics and Environmental Effects

PMTW usually refers to the comprehensive PMTW in many papers. In fact, PMTW from different stages has different characteristics during pickled mustard tuber production. In this review, PMTW's characteristics were measured through the standard measuring methods of wastewater contaminants, and by using other information such as the PMTW generation, discharge, environmental effects were obtained through field survey, governmental reports, questionnaires and references.

2.1. Pickled Mustard Tuber Wastewater Generation

PMTW is mainly produced in the pickling process of the fresh mustard tuber vegetable using NaCl and in the post-treatment process of washing, desalination, and dewatering. The production of mustard includes three submerging stages and three squeezing stages, as displayed in Figure 1. Specifically, the fresh mustard tuber is stacked in tanks and pickled with NaCl (first pickling). After the first pickled stage, saline wastewater with a salinity of 3–4 wt.% NaCl is produced. The second pickled stage (second pickling) and the third pickled stage (third pickling) were separated in the traditional process. However, these two stages are integrated as a pickled stage for the modern process duo to the development of new preservation techniques. The salinity of pickled wastewater is 12–13 wt.% NaCl usually. After the third pickled stage or the integrated pickled stage, the post-process of pickled mustard tuber requires a large amount of freshwater for washing, desalination, dewatering, and sterilization. The process to convert fresh mustard tuber to pickled mustard tuber requires an input of 0.17 t NaCl, and releases about 5 m^3 saline wastewater with 3.5 wt.% NaCl for one ton of mustard tuber product. Moreover, pickled mustard tuber generation is seasonal, so the discharge of hypersaline wastewater is intermittent. In the harvest season (at the end of February and beginning of March) every year, saline wastewater of the first pickled stage is huge, bringing great challenges for biological treatment and storage. The sequencing process of the second and third pickled mustard tuber stage lasts the whole year. So, the production of hypersaline wastewater of mustard tuber depends on the market requirement for pickled mustard tuber products.

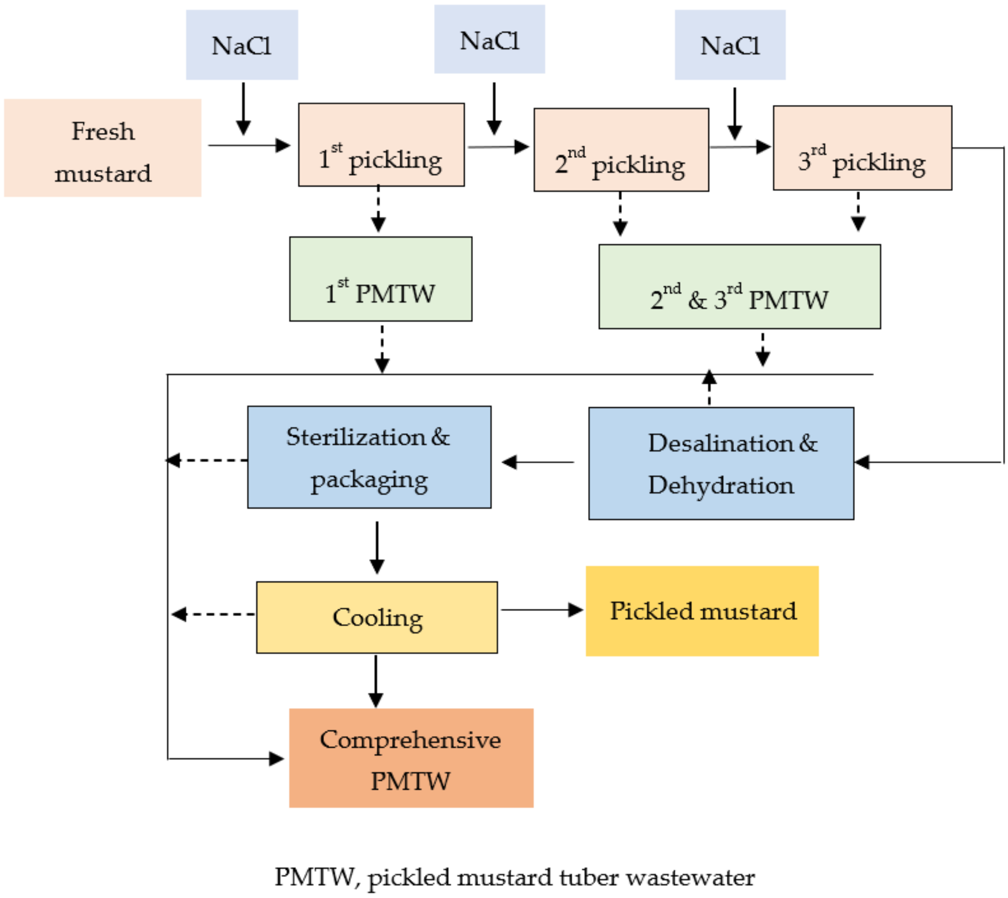

Figure 1. The schematics of pickled mustard processing and hypersaline wastewater generation.

2.2. Pickled Mustard Tuber Wastewater's Characteristics

The typical characteristics of PMTW are summarized and compared in Table 1. The concentrations of COD, NH_3-N, and Cl^- of the first pickled wastewater are 27,800–38,400 mg L^{-1}, 264–461 mg L^{-1} and 30,300–78,300 mg L^{-1}, respectively. The concentrations of COD, NH_3-N, and Cl^- of the second and the third pickled wastewater are much higher than the first pickled wastewater. Since fresh wastewater is used to desalt pickled mustard tuber, the contents of COD, NH_3-N, and Cl^- of comprehensive mustard wastewater are 7000–30,000 mg L^{-1}, 181–545 mg L^{-1}, 7370–33,200 mg L^{-1}, which are much lower than that of the pickled stages. The comparison of these stages of pickled wastewater demonstrates that pickled mustard wastewater has a high-level of NaCl concentration, but is also rich in organics and nutrients (N, P). The differences are attributed to the manufacturing process of the pickled mustard tuber.

Table 1. Characteristics of pickled mustard tube.

Pickles	Sources	COD	NH_3-N	TN	TP	TS	Cl^-	pH
		mg L^{-1}						
Mustard tuber	1st pickling	27,800–38,400	264–461	1600–2080	263–354	49,600–52,600	30,300–78,300	5.9–6.7
	2nd pickling	29,300–44,300	363–565	1620–2500	227–364	65,650–77,650	14,700–71,900	4.4–5.4
	3rd pickling	26,200–90,400	307–914	1640–3150	213–407	68,150–68,650	29,700–104,000	3.7–6.6
	Comprehensive	7000–30,000	181–545	210–2160	34–281	28,635–31,950	7370–33,200	4.6–5.5

2.3. Pickled Mustard Tuber Wastewater's Environmental Effects

There are various impacts of saline wastewater on environmental systems, including influences on crops, deterioration of water quality, soil infertility and ecological systems. In the present scenario, it is reported that dehydration caused by hypersaline wastewater restricts nitrogen uptake, which is important for crop growth [13,14]. Saline wastewater for crops with no treatment/management system leads to soil salt accumulation due to evapotranspiration. Na^+ and Cl^- compete with essential ions such as K^+, $NO3^-$, and $H_2PO_4{}^-$ for binding sites and transport proteins in root cells, causing a nutrient imbalance in plants [15].

The presence of excess salt in the soil leads to degradation of soil quality in terms of physical and chemical properties, poor groundwater quality and poor plant growth, which affects the farmer's economy. Generally, a higher accumulation of salts leads to structural damage to soil and the dispersion of clay minerals and particles is a major cause of reduced hydraulic conductivity and pore clogging. It further causes a thin crust at the soil's surface, resulting in water infiltration leading to reduced soil productivity and reduced crop yields [16].

In a study from Hunter River in New South Wales, the indiscriminate discharge of saline wastewater from different sources (drainage, coal mines and power plants) to the natural water bodies deteriorated the water quality and aquatic diversity [17]. Due to the surface runoff of saline wastewater, drinking water is affected, resulting in an increase in sodium and sulfates in the freshwater, thus implying community health and socio-economic implications [18]. Moreover, many floras and fauna of different ecological systems are impacted by the saline wastewater discharge, disturbing different morphological parameters, such as cell production, nutrient solubilization, etc. [19]. The salts in the saline wastewater further promote the flocculation of smaller particles, restrict the entrance of light to the water bodies and cause the water body's eutrophication [20]. In addition, saline wastewater reduces soil permeability resulting in the formation of flood areas.

As conventional biological wastewater treatment has difficulties in salt removal, human-induced salinity to the environment is the major environmental issue of PIWW. When saline wastewater is discharged into the environment, it can cause land salinization, pollute freshwater and harm aquatic lives [21]. Effects on agriculture are a significant concern for saline wastewater. A high salt concentration in the water and soil will negatively affect the crop yields and degrade the land. Increasing salinization on a global scale is decreasing average yields for most major crop plants [22]. This study shows that the yield of lettuce and Chinese cabbage starts to decline when the salinity level is 0.9 and 1.5 dS m^{-1}, respectively [23]. Extensive brine discharge threatens freshwater aquatic life in rivers and lakes and has the potential to heavily affect marine species [24]. A sudden increase in salinity influences the reproduction of marine species, consequently affecting their development and growth rate.

Considering the hazard of hypersaline wastewater, many countries and regions are starting to pay attention to the environmental effects of saline effluent. The prevailing international practice is to set emission limits for total salt in accordance with the environmental situation, in which they are affected. In addition, directive 2000/60/EC of the European Union requires member states to set standards related to effluent salinity for inland freshwater [25]. Therefore, sustainable agriculture and soil fertility management require appropriate and skilled management and treatment of saline wastewater prior to application to alleviate such problems.

3. Pickled Mustard Tuber Wastewater Treatment

3.1. Physicochemical Treatment

Physicochemical treatment plays an essential role in hypersaline wastewater treatment. The physicochemical processes mainly include thermal desalination, chemical precipitation, electrochemical oxidation-based technology, and membrane technology, as summarized in Table 2.

Table 2. Hypersaline wastewater treatment of pickled mustard tuber.

Process	Wastewater Source	Salinity (%)	Max. Cl^{-1} Tolerance (mg L^{-1})	Halophilic Bacteria	Module	Objects	Contaminant Removal	Reference
Bio-ceramic moving bed biofilm reactor	Mustard tuber wastewater	0.5–2.0	51,840	No	Lab-scale with 45 L	Optimization of influential factors and tolerance of organic loading shock	64.71% COD and 58.12% NH_3-N	[4]
Membrane bioreactor system (MBR)	Mustard tuber wastewater	2.0	–	No	Lab-scale with 620 L	The feasibility of Compound-type MBR for Mustard tuber wastewater treatment	80% COD, 94.16% NH_3-N, and 33.94%TP	[5]
Biological rotating cage	Mustard tuber wastewater	0.6–0.83	–	No	Lab-scale	Microbial communities	93% COD, 99.13% NH_4^+-N	[6]
Anaerobic/partial nitritation/ANAMMOX process	Mustard Wastewater	12	–	No	Lab scale with12 L	Start-up of the combined anaerobic, PN, and anammox process in the treatment of mustard wastewater	89.7% COD 86.2% TN	[7]
A/O process	Mustard Wastewater	2.0	–	–	Practical engineering	Cand N removal	70–95% COD, 60–80% NH_3-N, 70% TP	Our survey
Anaerobic/Contact oxidation/CASS	Mustard Wastewater	1.5	–	–	Practical engineering	C and N removal	80–95% COD, 60–92% NH_3-N	Our survey
A^2/O process	Mustard Wastewater	1.2–2.0	–	–	Practical engineering	C and N removal	90–95% COD, 80–90% NH_3-N	Our survey
Hydrolysis-acidogenesis-SBR-coagulation	Mustard tuber wastewater	1.0	–	Lab scale with 56 L	Optimizing conditions of combined technique for mustard tuber wastewater treatment	96% COD, 85.03% SS, 84.9% NH_4^+-N and 95.32% TP	[8]	

Table 2. *Cont.*

Process	Wastewater Source	Salinity (%)	Max. Cl^{-1} Tolerance ($mg\ L^{-1}$)	Halophilic Bacteria	Module	Objects	Contaminant Removal	Reference
Biological-chemical	Mustard tuber wastewater	7–7.5		Lab scale with 3.5 L	Treating mustard tuber wastewater with high salinity, high phosphorus, and high nitrogen.	56.6% COD, 20.8% NH_3-N, and 22% TP.	[9]	
Coagulation, anaerobic and electrode-SBBR integrated process	Mustard tuber wastewater	–	14,780	Lab scale	Find the optimal parameters to improve the performance of the combined system for mustard tuber wastewater treatment	83.26% COD, 70.98% TN, 52.56% TP	[10]	
Anoxic-oxic biofilm-membrane bioreactor	Mustard tuber wastewater	10	–	Pilot-scale with 630 L	Developing an optimal condition to obtain the highest treatment efficiency at lowest membrane fouling rate	90.3% COD, 92.4% NH_3-N, 61.6% TN and 98.1% SS	[11]	
Combined process of aerobicmicro-electrolysis-electrochemical oxidation-sedimentation	Mustard tuber wastewater	20–30	–	Lab scale	Parameter optimization to obtain the highest treatment efficiency	90.96% COD, 100% NH_3-N, 72.3% TN and 100% TP	[12]	

3.1.1. Thermal Desalination

Thermal desalination accounts for approximately 50% of desalination technologies [26], and multi-stage flash (MSF) distillation and multi-effect distillation (MED) are two commonly used thermal desalination processes. MSF using vaporization through multi-stage chambers produces high-quality freshwater with a little TDS (<30 ppm) [27]. Its maximum capability of flash water generation of MSF can be up to 75,000 $m^3\ d^{-1}$ for each unit, and the energy requirement is 20–500 kWh m^{-3} [28]. MED is the oldest desalination method to produce distilled water [29]. Awerbuch mentioned that the capability of freshwater generation could reach 45,400 m^3 per day [30]. Mechanical vapor recompression (MVR) was used to treat pickled wastewater. The inflow wastewater contained 8% NaCl and the outflow wastewater contained 20% NaCl. The recovered salt was reused for pickling the fresh mustard tuber. Although thermal desalination has many attractive merits such as zero liquid discharge, salt and minerals recovery, high quality of freshwater, etc., some challenges like expensive costs, high energy consumption, dry solid waste precipitates, and corrosion are faced when thermal desalination is scaled up [31].

3.1.2. Chemical Precipitation

Chemical precipitation for hypersaline wastewater of pickles mainly refers to coagulation and flocculation [32]. Coagulation and flocculation are usually used simultaneously. Coagulants carrying opposite charges are added to hypersaline wastewater to neutralize the charges on dispersed non-settleable solids [33] and flocculation is a gentle mixing stage that increases the particle size from submicroscopic micro floc to visible suspended particles.

Tian and Zheng studied the characteristics and mechanisms of flocculation treatment by recycling the physical-chemical sludge from the flocculation process (PAC and lime) in the unit for sauce wastewater treatment [34]. Their experiments showed that the adsorption and sweep of the reused sludge neutralized charges via PAC and CaO reacted with certain carbohydrates to form precipitation to remove 44.2% COD on average. Liu et al. used CaO as a coagulant and PAM as a coagulant to treat mustard wastewater with the removal efficiencies of 36.54% COD, 52.03% TP and 97.85% turbidity, respectively. CaO and PAM could automatically adjust pH, reduce irritating odor and improve biodegradability [35]. In contrast, coagulation and flocculation have some main disadvantages, such as high cost, accurate dosing and frequent monitoring, which brings some inconveniences for hypersaline wastewater treatment [36].

3.1.3. Electrochemical Oxidation

Hypersaline wastewater is treated by electrochemical oxidation due to its high conductive capability. For example, Qu et al. used electrochemical oxidation to pretreat pickled hypersaline wastewater and obtained 55.74% COD and 99.77% ammonia removal [37]. They also used the same method to remove ammonia nitrogen from pickled hypersaline wastewater and discussed the factors of ammonia nitrogen removal. They found that ammonia removal was determined by time, current density, the inter-electrode distance, electrode plate area/water volume ratio and pH value. Based on these finds, they conducted the corresponding optimization and gained 99.94% of ammonia removal and 96 kWh/kg (NH_4^+-N) of energy consumption [38]. Sheng et al. employed a boron-doped diamond anode to oxidize organics of mustard tuber wastewater and achieved 80.4% COD removal and 100% ammonium removal rate with 45.8 kWh m^{-3} energy consumption [39]. However, high energy consumption, electrode break down, and concentration, limit the application of electrochemical oxidation. Some researchers focus on new technologies—such as advanced oxidation, which is a representative technology with low energy input. Moraes et al. applied photo-Fenton oxidation to treat saline wastewater containing hydrocarbons [40]. Their experimental results demonstrated that photo-Fenton oxidation was feasible to treat high concentrations of salt of 2 g L^{-1}. In contrast, the concentration of residual iron after the photo-Fenton reaction was above the concentration accepted by the environment, which required expensive technologies to reduce it to an acceptable level [41]. Hence, photo-Fenton oxidation has not been a general approach for practical engineering yet.

3.1.4. Membrane Technology

Membrane technology such as reverse osmosis, nano-filtration, ultra-filtration and microfiltration, electrodialysis and electrodialysis reversal is the most commonly used for hypersaline wastewater treatment [42,43]. Scholz and Lucas conducted a technic and economic evaluation of membrane filtration and their results indicated that membrane filtration enabled a 90% recovery rate of pickled agents since ion concentration and separation do not require extra chemical input or thermal energy [44]. Vaudevire and Koreman designed a pilot-scale study on the loop of NaCl use to reduce disposal towards a zero liquid discharge. The results demonstrated that nanofiltration membrane had 87% of DOC, 85% of sulfate, 80% of Na^+ and 100% Cl^- retention [45]. These studies suggest that membrane technology has merits for water recovery, a large capability of ion separation and economic costs for salinity reduction associated with secondary effluent (or other wastewater sources) TDS levels.

The costs of different processes within the pickled mustard tuber process depend on the original salinity. As displayed in Figure 2, the costs of electrodialysis has increased from USD 4.5 to USD 5.5 for the effluent with 0.5 wt.% NaCl and from USD 5.0 to USD 6.5 for the effluent with 0.1 wt.% NaCl, respectively, with the NaCl concentration of the influent escalating. On the contrary, the cost of MVR decreases to USD 4.5 when the effluent NaCl concentration increases to 15%. Besides, the cost of electrodialysis depends on the

NaCl concentration of the effluent. Even if the lowest cost of electrodialysis is USD 4.5, it is much more expensive than that of a biological process at USD 1.5. Additionally, membrane fouling, acid-base resistance and corrosion are great challenges for electrodialysis. Therefore, proper uses of physical-chemical and biological treatments can make the effective treatment of PIWW come true. The above research also mentioned the limitations, such as pre-treatment, solids removal, membrane fouling and concentrated and waste stream disposal. Therefore, physical-chemical treatment displays excellent performances in salt removal, but poor efficiency for organic, nitrogen and phosphorus.

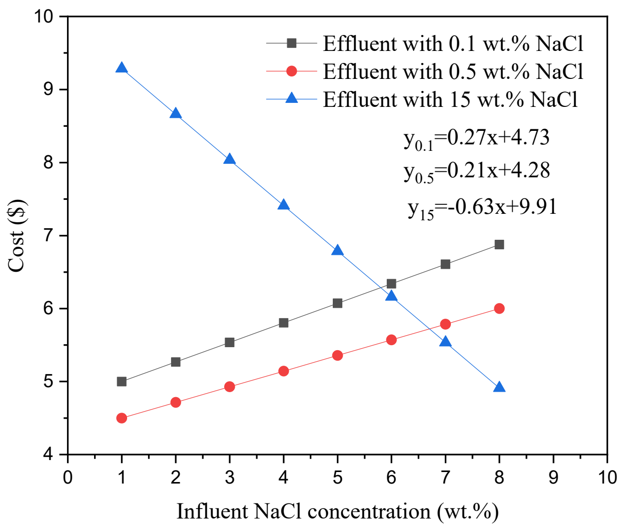

Figure 2. The costs of electrodialysis under different influent NaCl concentration.

3.2. *Bio-Treatment of Pickled Mustard Tuber Wastewater*

Although NaCl detrimentally impacts the activity of the microbial community, biotreatment is the mainstream of PMTW treatment and its process is shown in Figure 3 [46–50]. The following section discusses aerobic oxidation, anaerobic digestion and aerobic/anaerobic hybrid treatment for hypersaline wastewater, as summarized in Table 2.

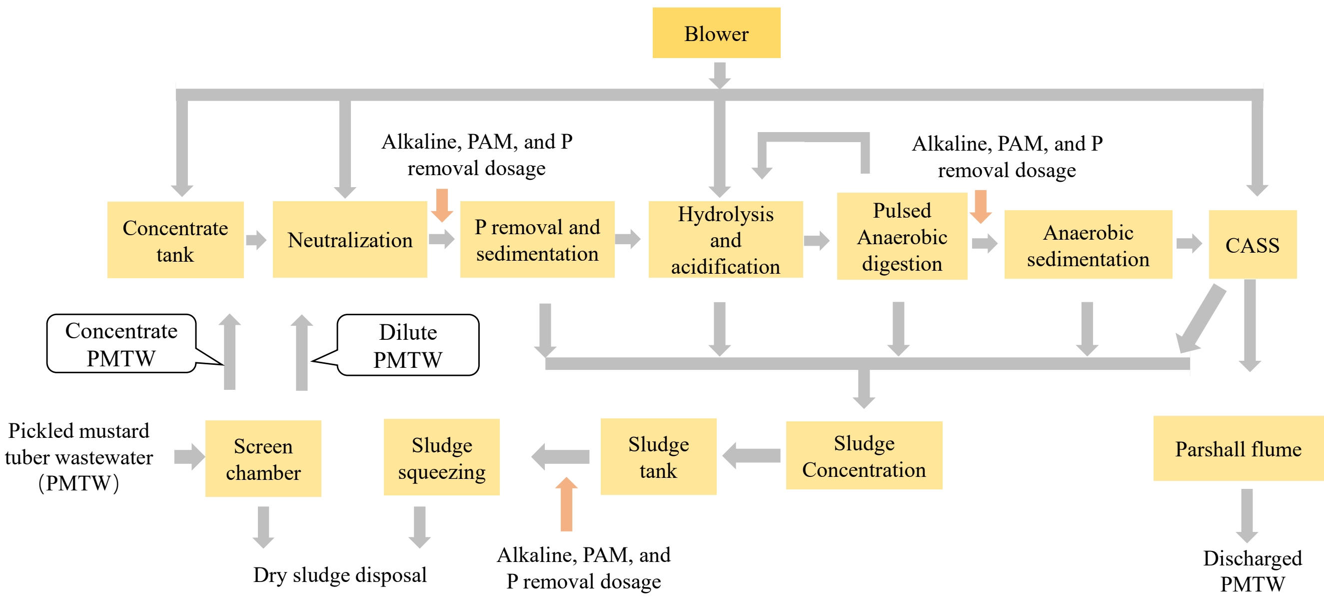

Figure 3. Schematic diagram of pickled mustard tuber wastewater.

3.2.1. Aerobic Oxidation

The feasibility of aerobic oxidation to remove carbonaceous, nitrogenous, and phosphorous pollution from high salt concentrations has been studied for decades [50,51]. The current interest is focused on aerobic granules formation and the efficient aerobic treatment processes. Aerobic granular sludge (AGS) is a type of sludge that can self-immobilize flocs and microorganisms into spherical and strong compact structures [52,53]. The formation of sludge granules is slow but can become a salt-tolerant granular under the high-salinity condition [54]. The formation mechanism of AGS has been discussed for some decades. The first one of note is the crystal nucleus hypothesis. As discussed in Long's report, when SBR is inoculated with aerobic (anaerobic) granules, an inorganic material, or some inorganic salts, the system as a crystal nucleus assists the formation of aerobic granular sludge through microbial growth [55]. Tao's research supported this hypothesis, that granular activated carbon provided the interaction media for sludge to attach and enhance the morphological regularization of sludge [56]. Verawaty et al. also found that flocs attachment to the surface of the seeding granules reduced biomass washout during granulation [57].

The second of note is the selection pressure hypothesis. The hydraulic selection pressure and biological selection pressure are used to optimize the operation parameters and explain aerobic granular sludge formation. The salt concentration is not only hydraulic selection pressure, but also biological selection pressure. As the hydraulic selection pressure, the stepped increase in salt concentration from 0 wt.% to 9 wt.% resulted in bigger flocs so that young granules could stay in the sequencing batch reactors (SBR) system, which could be seen as the enhancement of the function of decreased settling time [58]. As a natural selection pressure, elevated salinity alters the microbial community to adapt to hypersaline stress. According to Lim's study, the increase in salinity contributed to an increase in the halotolerant bacteria, thus making the microbial community tolerant of different salinity levels. As a result, some adapted bacteria were capable of salt tolerance, while other unadaptable bacteria were granularly washed out or weakened [59].

The third hypothesis is described through the DLVO theory. The double-layer compressed with salt concentration increasing from 0 wt.% to 9 wt.%. The surface potential was reduced, but the surface charge density kept constant, which reduced the total repulsive forces between different zooglea, and enhanced the maturity of young granules [60]. The addition of seawater significantly accelerated the granulation process and the salinity-induced decrease in the electrostatic charge on the surface of cells allowed sludge flocculation [61]. Thereby, salts are somewhat beneficial for the aggregation of flocs and the maturation of salt-tolerant granular sludge.

More efforts have been focused on the optimizations of physical factors to resist adverse influences and achieve the best performances for hypersaline wastewater treatment, such as pH value, salinity, substrate loading rate, solids retention time, dissolved oxygen, cycle time/hydraulic retention time, seed sludge, and reactor configuration [62]. Some researchers have attempted to adapt conventional micro-organisms to high salinity [48]. Nevertheless, it has been proved that halophilic inoculum is the best way to improve the performance of the aerobic treatment processes [50].

For aerobic oxidation, the frequently mentioned processes include the AGS, physical contact oxidation, biofilm, and rotating biological cage, as summarized in Table 2. In these processes, starting a bio-ceramic moving bed biofilm reactor for the hypersaline wastewater treatment of pickled mustard tuber is worth paying attention to. Under the optimal conditions of organic loading rate (OLR) of 3.3 kg COD m^{-3} d^{-1} and operating temperature of 25 °C, the biological contact oxidation process showed the highest salinity tolerance of 51.84 g L^{-1} [63]. Furthermore, the composite membrane bioreactor system with intermittent aeration to treat brine wastewater of pickled mustard tuber is recommended. At the steady status, the reactor was filled with hypersaline wastewater characterized to 320–580 mg L^{-1} COD, 106–190 mg L^{-1} NH_3-N, and 27–45 mg L^{-1} PO_4^{3-} and gained the corresponding removal efficiencies of 66%, 94%, and 34%, respectively [64]. The literature indicates that only a few processes use single aerobic oxidation due to a large amount of energy for aeration and generate excess sludge bio-solids or sludge. Wang et al. studied the multiple potential pathways and key microorganisms of nitrogen removal in a modified sequencing batch biofilm reactor treating hypersaline mustard tuber wastewater [65]. Their results indicate that the enrichment of particular halophilic functional bacteria with multiple nitrogen removal pathways is a good idea for the efficient treatment of high-concentrated hypersaline industrial wastewater.

3.2.2. Anaerobic Digestion

As the global concept of energy-saving and sustainable recovery of wastewater becomes increasingly popular, anaerobic digestion experiences rapid development. Anaerobic treatment can endure high organics loading, consumes low energy, and generates methane to be used to treat hypersaline wastewater [66,67]. The core of AD technology in anaerobic granular sludge (AnGS) is composed of hydrolytic fermentation bacteria, acid-producing bacteria, and methanogenic bacteria under high hydraulic shear. The structure formed by different species is a micro-ecosystem with symbiotic or symbiotic relationships, conducive to providing physiological conditions for microbial growth and maintaining a relatively stable microenvironment.

It is believed that AnGS granulation is mainly divided into three stages: (1) the process of attracting adhesion between bacteria and substrate; (2) the formation of microbial aggregates; (3) the increasing size of mature sludge. The attracting adhesion process between bacteria and substrate is the initial stage of AnGS formation and an important stage in determining the structure of AnGS. For example, Lettinga studied the potential causes of AnGS granulation and believed that the slow-growing bacteria and extremely hungry microorganisms (methane-producing, acetic acid bacteria, etc.) produced microbial aggregates and they even achieved an aggregated state and thus formed a balanced ecosystem for the benefits in pursuit of each other [68]. AnGS can retain a high concentration and withstand a high organic matter volume load, even if it confronts with a high gas production rate and upward flow rate [69].

Under high salinity, Ca^{2+} is considered to have a positive effect on the process of AnGS granulation and can increase the strength of the particles [70,71]. Pevere et al. compared the effects of Ca^{2+} and Na^+ on AnGS aggregation. They found that Ca^{2+} strongly changed the viscosity of AnGS suspension, enhanced the physicochemical interaction between small anaerobic particles, and induced the formation of larger particles. At the same time, Na^+ had a slight effect on the fine particle viscosity of AnGS suspension but will reduce the strength of AnGS once the concentration of Na^+ was excessively high [72,73]. Jeison et al. found that the high salt of NaCl led to a reduction in particle strength, making the process unstable during the long-term operation of the reactor and the sensitivity of granular sludge to Na^+ concentration decreased. As such, 7 g Na/L concentration wastewater can replace the role of calcium in the particles, which means that bacteria have adapted to the high salinity.

For anaerobic digestion, interspecies exchange of electrons are considered important in diverse environments, as they enable microbial communities to gain energy from reactions. There are two widely recognized mechanisms for microbial electron transfer [74,75]. The first mechanism is the mediated interspecies electron transfer [76,77]. As illustrated in Figure 4, H_2 and formate are important electron transfer molecules in various methanogenic environments. H_2 is a powerful electron donor under anoxic conditions. Although less than one-third of methane production depends on hydrogenotrophic activities, the utilization of H_2 and its interspecies transfer is critical since H_2 limits the rate. Furthermore, the pressure of H_2 controls the extent of H_2 producing reactions, so H_2 must be continuously consumed by partner organisms for the syntrophic interaction to occur [78]. Other than H_2, formate detected in most methanogenesis systems has proved to be an important extracellular electron carrier in syntrophic metabolism as well. The redox potential of formate/CO_2 ($E_0{}' = -0.432$ V) is close to that of H_2/H^+ ($E_0{}' = -0.421$ V), indicating that formate and H_2 have the same niche as an intermediate. However, the solubility, transfer speed, and diffusion distance of formate are much higher than H_2 in syntrophic propionate and butyrate degradation [79].

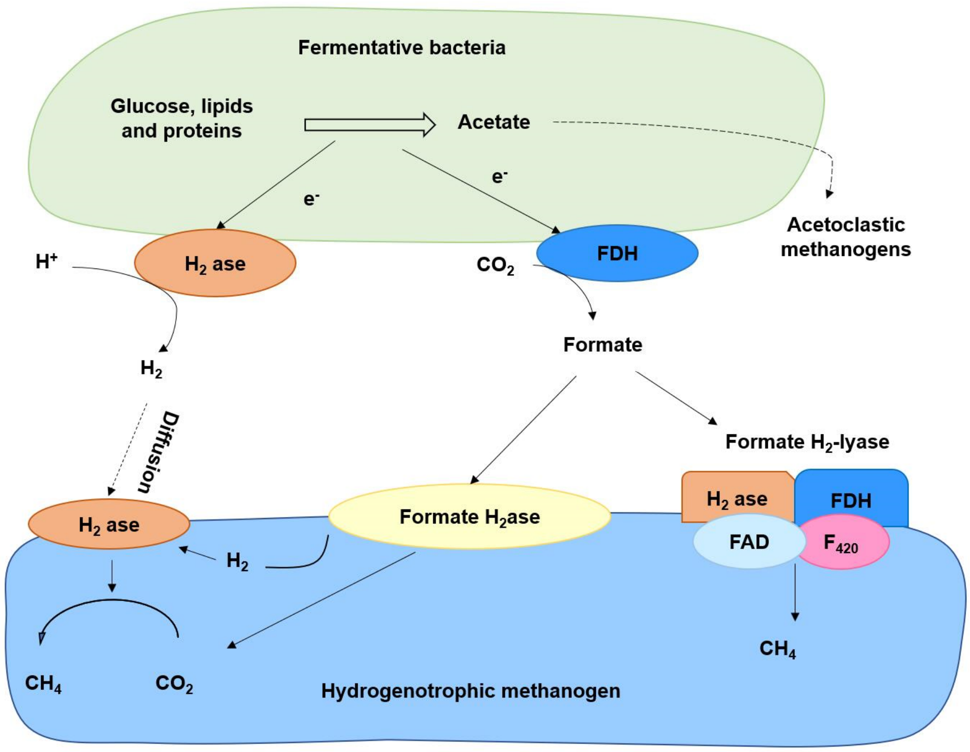

Figure 4. The mediated interspecies electron transfer. Reprinted with permission from ref. [1]. Copyright 2016 Copyright Elsevier.

Another mechanism is direct interspecies electron transfer (DIET), as displayed in Figure 5. DIET is a syntrophic metabolism, in which the free electrons flow from one cell to another without being shuttled by reduced intermediates like hydrogen or formate. Potential DIET mechanisms include electron transfer via electrically conductive pili, electrically conductive materials, electron transport proteins, and the diffusive exchange of electrons between species through soluble electron shuttles such as H_2 [80,81]. The electron exchange between syntrophic partners together by DIET requires cells to develop efficient conductive contacts via pili and cytochromes. Based on thermodynamic principles, when the degradation of propionic acid is coupled with methanogenesis, the change of Gibbs free energy (ΔG) is +3 kJ mol^{-1}, indicating that the reaction is not spontaneous. However, with the presence of sulfate-reducing bacteria, ΔG becomes -180 kJ mol^{-1}, demonstrating that the conversion of propionic acid to methane is more favorable [78,82]. In other words, the salt in the system has a specific promotion effect on methane production.

Some anaerobic processes have been developed from laboratory tests to practical engineering applications, as summarized in Table 2. Anaerobic sequencing batch biofilm reactor (ASBBR), a new and efficient reactor, was applied to treat most organic compounds in mustard tuber wastewater. The results demonstrated that under 30 °C, there was a draining ratio of 1/3, and two-day hydraulic retention time, the biofilm density of 50%, and the maximum removal of COD reached 90.5% [83]. In contrast, high salinity and low temperature inhibit the activity of anaerobic microorganisms and lead to low treatment efficiency for ASBBR in winter. To solve this problem, betaine was added to the reactor to improve the activity of the anaerobic sludge. Under the optimal dosage of betaine of 0.5 mmol L^{-1}, the dehydrogenase activity of anaerobic microorganisms and the COD removal efficiency was increased by 18.6% and 18.1%, respectively [84]. Chen et al. combined an anaerobic sequencing batch biofilm reactor, a sequencing batch reactor (SBR), and an upflow anaerobic sludge blanket (UASB) for anaerobic treatment, partial nitridation and anammox using high salinity wastewater from the mustard pickling industry as the substance. The results demonstrated that 89.7% of COD and 86.2% of nitrogen were removed, respectively, under high salinity of 12.0 g NaCl L^{-1} and nitrogen loading rate of 258 mg $(L\cdot day)^{-1}$. However, the dilemmas of this process included a slow growth rate, slow granulation, the unstable and poor removal efficiency of contaminates, vulnerability to contaminants, and disintegration of anaerobic granular sludge.

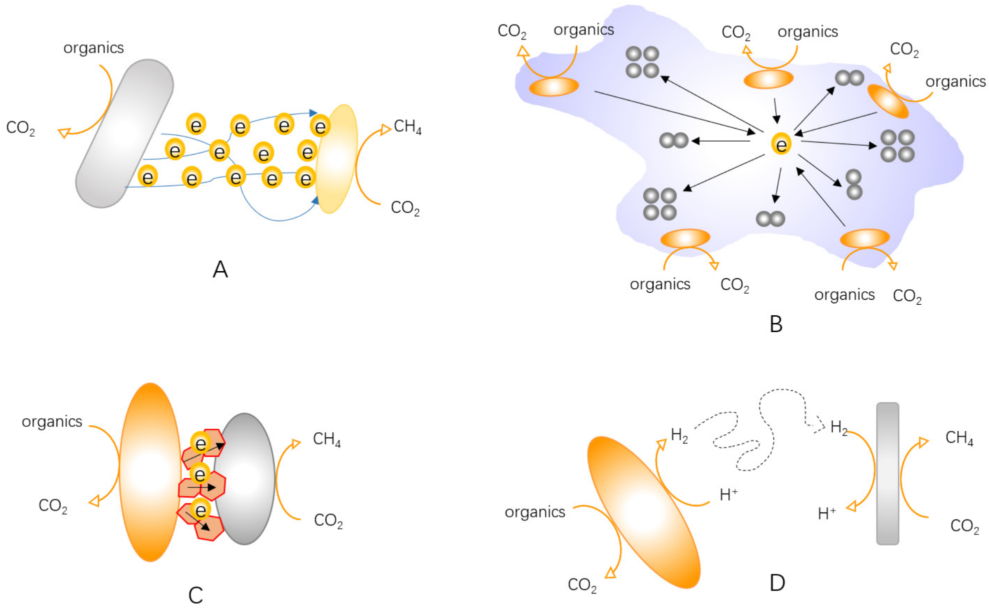

Figure 5. Mechanisms for interspecies electron transfer with emphasis on DIET. (**A**) electron transfer through electrically conductive pili, (**B**) electron transfer through electrically conductive materials, and (**C**) electron transfer between electron transport proteins associated with outer cell surfaces. (**D**) electron contrasts with the diffusive exchange of electrons between species through soluble electron shuttles such as H_2. Reprinted with permission from refs. [2,3]. Copyright 2013 Copyright Elsevier.

3.2.3. Combined Anaerobic and Aerobic Treatment

A single aerobic or anaerobic technique has not yet been sufficiently effective in treating hypersaline wastewater. Many studies combined aerobic and anaerobic processes for brine wastewater treatment to enhance the treatment efficiency. In Table 2, we discuss the combined anaerobic and aerobic techniques. Lefebvre et al. designed a system combining the anaerobic digestion of tannery soak liquor and an aerobic post-treatment. The combined anaerobic/aerobic treatment system removed 96% of COD after one month of the operation under an OLR of 0.5 kgCOD m^{-3} d^{-1}, HRT of 8 days and the concentration of total dissolved solids of 71 g L^{-1} [85]. Apart from carbonaceous pollution removal, the anaerobic/aerobic combined processes could also eliminate biological nitrogen, phosphorus, and other pollutants from saline wastewater. Chen et al. established a combined anaerobic/partial nitrification/anammox process to treat high salinity mustard wastewater. After anaerobic sequencing batch biofilm reactor (ASBBR), sequencing batch reactor (SBR) and upflow anaerobic sludge blanket digestion (UASB), the removal rates of COD and NH_3-N were 89.7% and 86.2%, respectively, when the inflow containing the salinity was about 16.1 g NaCl L^{-1} [86]. Dong et al. optimized the performance of saline MTWW treatment concerning the removal efficiencies of COD, ammonia nitrogen (NH3-N), and total nitrogen (TN) through a pilot-scale packed cage rotating biological contactor system. They obtained the optimal region for energy consumption and maximum COD, NH3-N, and TN removal efficiency with an ORL of 26.71 kg day^{-1}, RDV of 1.62, and IR of 46% [87]. According to our statistical data, the costs of bioprocessing is between USD 1.5–2.5 per ton, but it does not remove any salts from PMTW.

3.3. Physicochemical and Biological Hybrid Arts

To improve the efficiency of hypersaline wastewater treatment, the combination of aerobic oxidation and anaerobic treatment or integrated physical-chemical processes has been paid increasing attention in recent years as combined processes can facilitate advantages and overcome the disadvantages [88,89]. The integrated processes of PMTW treatment are summarized in Table 2.

The combination of physical-chemical and biological treatments shortens the treatment period and enhances more complete removal of organic contaminants than sole biological or physical-chemical technology, which is very promising for treating hypersaline wastewater [90,91]. The recent research on this topic is summarized in Table 2. Wu et al. studied a combined hydrolysis-acidogenesis-SBR-coagulation process in the laboratory scale for mustard wastewater treatment and found that the removal rates of COD, SS, NH_3-N, and TP reached 96%, 85.03%, 84.9%, and 95.32% under the conditions of 22 h HRT, 300 mg L^{-1} PAC and 6 mg L^{-1} PAM, respectively. Yin et al. conducted a study on comprehensive mustard wastewater treatment using the combined process of aerobic micro-electrolysis-electrochemical oxidation-sedimentation. The removal rates of COD, BOD_5, NH_3-N, TP, SS and salinity were 90.96%, 70.88%, 100%, 100%, 91.6% and 26.34%, respectively [92]. Chai et al. optimized the operating parameters to minimize membrane fouling for mustard tuber wastewater treatment in an anoxic-oxic biofilm-membrane bioreactor [62].

Scholz and Lucas used membrane filtration of reverse osmosis and ultra-filtration to reuse the tanning chemicals that enabled a 90% recovery rate and topped this up with 4% salt (NaCl) [44]. Vaudevire and Koreman proposed a pilot case of ion exchange membranes aimed at the close loop of NaCl to achieve zero liquid discharge. The nanofiltration membrane retained dissolved organic carbon of 87% and sulfate of 85% and allowed Na^+ and Cl^- passing with removal efficiencies of 80% and 100%, respectively [45]. Electrodialysis is another significant technology for seawater desalination [93,94], but little related work was reported in publications for PMTW. Physicochemical and biological hybrid art is the most suitable process from the cost perspective, which is approximately USD 6.00 per ton. Although this cost is higher than bio-treatment, salts and main organic contaminants can be removed simultaneously.

4. Pickled Mustard Tuber Wastewater's Reuse

4.1. Nutrient Recollection

Nutrient recovery from hypersaline wastewater deserves special attention, and related studies have been reported by many scholars [95]. Wang et al. carried out an investigation on phosphorus recovery from mustard tuber wastewater and achieved 99% phosphorus recovery by integrating biological and chemical processes [96]. Gao et al. investigated the conditions affecting the precipitation of calcium phosphate for recovery from wastewater and found that pH control and the initial Ca/P molar ratio change could increase the precipitation efficiency through the batch tests [97]. Xie et al. reported that membrane-based processes could recover nutrients from saline wastewater [98]. These studies indicate that there is nutrient recovery potential from hypersaline wastewater, although there is still a large gap between lab-scale research and practical applications.

4.2. Acids and Alkaline Regeneration

Na^+ and Cl^- are important sources of NaOH and HCl. The salt of mustard tuber wastewater can be repurposed for acid and caustic production through multiple electrolysis cycles. Bipolar membrane electrodialysis is a friendly technology. Ibáñez et al., who studied acid and base recovery from softened reverse osmosis brine concluded that bipolar membrane electrodialysis presented the production of 1.0 M or higher concentrated acid and base with current efficiencies in the 60–90% range [99]. Thiel et al. reviewed many methods for producing sodium hydroxide from seawater reverse osmosis and found that bipolar membrane electrodialysis showed the best potential to meet the techno-economic requirements [100]. Our study found that the maximum desalination, acid and alkaline generation rates of bipolar membrane electrodialysis for pickled mustard tuber were 0.304 mol h^{-1}, 0.114 mol h^{-1}, and 0.136 mol h^{-1}, respectively [101]. The brine, seawater and pickled mustard tuber results signify that the acid and alkaline regeneration is a good approach to salty wastewater reuse.

4.3. Renewable Energy Recovery

Energy consumption is a widely concerned topic for hypersaline wastewater treatment. Some researchers study energy saving. Chen and Yip developed an innovative cascading osmotically mediated reverse osmosis technology to eliminate the disadvantages of conventional reverse osmosis, which achieved up to about 33% energy saving [102]. Boo et al. studied temperature swing solvent extraction for selective extraction of water over salt from a saline feed of 1.5 M NaCl [103]. The other aspect is sustainable energy recovery. Ansari et al. introduced a hybrid system of halotolerant organisms in forwarding osmosis based anaerobic digestion. This system produced biogas and simultaneously allowed the forward osmosis system to reduce salt concentration [104]. Kim and Logan applied a new bio-electrochemical system by supplying additional voltage to remove salts and organic matters under the concentration of 8 g NaCl L^{-1}, of which they gained removal efficiencies of salts and COD of 84% and 94%, respectively. Hydrogen production was achieved with a maximum production rate of 3.6 m^3-H_2 m^{-3} -electrolyte per day at an applied voltage of 1.2 V [7]. Guo et al. utilized mustard tuber wastewater as a fuel for a typical dual-chamber microbial fuel cell. Microbial fuel cells had an internal resistance of 121 Ω, a Columbic efficiency of 67.7 ± 1.0%, a maximum power density of 246 mW m^{-2}, and a maximum COD removal rate of 85%, respectively [3]. Our latest paper reported electricity generation and acid and alkaline recovery from pickled waters/wastewaters through anaerobic digestion, bipolar membrane electrodialysis and a solid oxide fuel cell hybrid system. The results demonstrated that AD converted 70% of chemical oxygen demand (COD) to biogas with 0.051 L-CH_4 g-COD^{-1} on average. SOFC used recovered biogas and NH_3/H_2 and output 500 mW cm^{-2} and 530 mW cm^{-2} of peak power densities, respectively. The hybrid system finally gained 55% of maximum net energy efficiency for the third pickled water [101].

5. Pickled Mustard Tuber Wastewater's Prospects

Due to the agricultural industry's low profitability and broad scope, there are various difficulties in PMTW that are not widely reported. Therefore, how to find a technically feasible, realistically operable and easily accepted way to treat PMTW and promote the harmonious development of the mustard tuber industry and local society and economy has become a significant issue. According to our published work on AD-ED used to regenerate HCl and NaOH, the brine juice produced in the "three-pickling" processes output higher energy and brought lighter inhibition on bacteria when the brine juice was separately treated from the comprehensive PMTW. Our work indicates that the combined process is cost-effective for PMTW treatment, which is primarily required for a long time in the future. Mustard tuber sauce production may be one choice since the brine juice produced in the pickling process of fresh mustard tuber contains various sugars, amino acids and nutrients. The development of mustard tuber sauce can reduce the discharge of PMTW and the consumption of soybeans and NaCl. Moreover, sustainable reuse of PMTW is to be an essential prospect for PMTW.

6. Conclusions

This manuscript reviewed the characteristics, existing and emerging technologies, and prospects of PMTW treatment in the future. PMTW features concentrated NaCl of 7 wt.%, COD of 30,000 mg L^{-1}, NH_3-N of 400 mg L^{-1}, and TP of 300 mg L^{-1} on average, affecting agricultural crops, water quality deterioration, soil infertility and ecological systems. PMTW is mainly treated by physicochemical processes (USD 17.00 per ton), biological processes (USD 2.00 per ton) and integrated both (USD 6.00 per ton). The difficulties with the PMTW results are that the research focuses on salt and organic contaminant removal at present, but PMTW sauce and sustainable reuse may be a bright prospect in the future.

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