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Review

Improving the lipid recovery from wet oleaginous microorganisms using different pretreatment techniques

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ABSTRACT

Lipid extraction directly from the wet oleaginous microorganisms for biodiesel production is preferred as it reduces the energy input for traditional processes which require extensive drying of the biomass prior to the extraction. The high water content (≥80% on cell dry weight) in the wet biomass hinders the extraction efficiency due to the mass transfer limitation. This limitation can be overcome by pretreating wet biomass prior to the lipid extraction using pressurized gas that can be used alone or combined with other pretreatments to disrupt the cell wall. In this review, an extensive discussion on different pretreatments and the subsequent lipid extraction using these pretreatments is presented. Furthermore, a detailed account of the cell disruption using pressurized gas (e.g., CO_2) treatment for microbial cell lysing is also presented. Finally, a new technique on lipid extraction directly from wet biomass using the combination of pressurized CO_2 and microwave pretreatment is proposed.

1. Introduction

Renewable biofuels are considered one of the main future transportation fuel sources which have been studied extensively as the petroleum-based fuels suffer from unsteady market price, adverse environmental effect, and finite reserves (Guo and Englehardt, 2015; Ramadhas, 2016). Biodiesel, one of the main components of renewable fuel portfolio is produced from the transesterification of oil (lipid) with an alcohol such as methanol in the presence of a catalyst where glycerol is produced as a byproduct (Kostić et al., 2016; Thanh et al., 2014). Biodiesel as an alternative fuel is particularly attractive because they are biodegradable, renewable, and environmentally benign (Yaakob et al., 2013). Traditionally, biodiesel is produced from vegetable oil (e.g., soybean) as well as from waste or used cooking oil, and animal fat (Marangoni, 2017; Sales et al., 2017; Tangy et al., 2017). Biodiesel obtained from vegetable oil is known as the first generation biofuels (Correa et al., 2017). Biodiesel from vegetable oil meeting ASTM D6751 or EN 14214 standards can be used to blend (e.g., B20, 20% biodiesel and 80% petroleum diesel) petroleum diesel to improve the fuel properties (Knothe et al., 2015). Though different properties of the biodiesel obtained from vegetable oil are comparable with the diesel fuel, the utilization of vegetable oil for biofuels faces a tough challenge from the debate "food vs. fuel" (Sut et al., 2016). Furthermore, vegetable oils have lower oil yield and higher land area requirement making them unrealistic in long-term goal (Rawat et al., 2013). Though biodiesel obtained from animal fat or waste cooking oil does not compete with food sources like vegetable oil, the high free fatty acid (FFA) content and additional treatments make the process uneconomical (Huang et al., 2010). Lignocellulosic biomass such as forest residue, rice straw, woody biomass, etc. can also be used as a feedstock for renewable diesel to replace the petroleum diesel, which is known as second generation biofuels (Ghosh et al., 2017; Moreno et al., 2017). Although the second-generation biofuels are a good source for replacing conventional petroleum diesel, the high production cost due to the technical barriers makes them uneconomical for the long-term solution and have not been proven on any significant commercial scale (Naik et al., 2010).

Different oleaginous microorganisms would be able to potentially resolve the issues that arise from both first and second-generation biofuels, and replace them for long-term future biofuel applications. Numerous research has been performed on biofuel production from different oleaginous microbes (Chatterjee and Mohan, 2018; Cho and Park, 2018; Mishra et al., 2018; Tang et al., 2018). Oleaginous microbes are microbes that can accumulate more than 20% lipid on dry cell weight (DCW) (Jin et al., 2015). Some of the unique advantages of using oleaginous microbes for producing biodiesel are a) they are not season dependent for their growth like the vegetable oil, b) their production rate is very high, and c) they are not used as food sources; thus, eliminating the food vs. fuel issue (Ryu et al., 2013). Another

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noteworthy advantage of using oleaginous microbes is their ability to utilize wastewater and municipal sewage sludge as their growth medium for lipid accumulation (Melero et al., 2015). In addition to different microbes, algae have been studied extensively due to their diversity, and it is reported that there are approximately thousands of algae species that can accumulate lipid on various DCW basis (Collet et al., 2014; Teymouri et al., 2018). Some of the algae species (e.g., *Botryococcus braunii*) can accumulate up to 75% oil on DCW (Amaro et al., 2011). Different oleaginous yeasts are also considered as potential candidates for biofuels production for their advantages such as the ability to use a wide range of substrates, faster production rate compared to algae, and tolerance of a wide range of pH for their growth (Leiva-Candia et al., 2014; Santamauro et al., 2014). Other than yeast and algae, many other microbes have also been investigated for their potential to produce lipids (Shields-Menard et al., 2018). Though different bacterial species are generally used for food preservation application (Gómez-Sala et al., 2016; Portillo et al., 2018), some of them are reported to accumulate lipid up to 80% on DCW, which can also be used for the biodiesel application (Alvarez et al., 1996).

Lipid extraction is the initial step for biodiesel production from oleaginous microorganisms. After the cell culture of microbes either in an industrial scale fermenter or in a shaker (small-scale fermentation), the grown cell suspension is concentrated to separate the lipid-containing solid cell pellets from the liquid portion (supernatant). The supernatant is either discarded or stored for other purposes. The concentrated cell pellets still contain more than 60% moisture (w/w basis), which is further dried in a freeze dryer or other means of drying to make the final solid biomass with more than 80% solid on the weight basis for lipid extraction (Jin et al., 2012; Willis et al., 2014). It is estimated that the overall process cost increases significantly if drying is added in the biomass processing prior to lipid extraction making the process economically unfeasible in large-scale applications (Reddy et al., 2014; Wu et al., 2017). Hence, the lipid extraction directly from wet biomass is preferred to eliminate the energy-intensive drying of the wet biomass (Yoo et al., 2012). Fig. 1 shows two different processes for the production of biodiesel at the laboratory scale; Fig. 1(a) depicts the lipid production by utilizing the drying of the biomass prior to the lipid extraction, and Fig. 1(b) presents the biodiesel feedstock production directly from wet microbes without drying. The energy-intensive drying of the biomass can be avoided by producing biodiesel via wet lipid extraction route. Though lipid extraction from wet biomass is preferred and a viable option to produce biodiesel on large scale, the presence of moisture in the wet biomass and cell wall impede the lipid recovery due to the increased mass transfer limitation. Hence, a form of pretreatment of wet biomass that can rupture the cell wall is preferred. The intracellular lipid comes out to the surrounding aqueous medium due to the cell disruption, which can be recovered using the common lipid extraction technique (i.e. using hexane). The microbial cell disruption is a useful technique to implement on wet biomass for improving lipid extraction for biodiesel production that can be an attractive process in renewable biofuels application.

There have been recent review articles on microbial cell disruption using both mechanical and non-mechanical methods for biofuels application (Lee et al., 2017; Show et al., 2015). Researchers also investigated the effect of pressurized gas treatment for cell disruption mainly for food preservation application (Hu et al., 2013; Morris et al., 2007). However, there is a lack of information in the area of cell disruption using pressurized gases for biofuels application including the effect of different factors on pressurized gas treatment, mechanism of the process, and the role of solubility of the treated gas in the lipid-rich microbial cell. Hence, the main objective of this review is to present the current scenario of lipid extraction for biofuel application using different pretreatment techniques primarily from wet oleaginous microbes. In our previous study, the research was mainly focused on upstream processing to maximize the lipid production utilizing different fermentation media (e.g., activated waste sludge and industrial wastewater), and different carbon (e.g., glycerol, glucose, wheat straw, and miscanthus) and nitrogen (e.g., N-acetylglucosamine) sources for the biodiesel production by cultivating both yeast and bacteria (Easterling et al., 2009; Mast et al., 2014; Paraschivescu et al., 2008; Revellame et al., 2013; Schneider et al., 2012; Shields-Menard et al., 2018; Taconi et al., 2008; Zhang et al., 2011). However, we put minimal emphasis on downstream processing such as pretreatment of wet cell biomass prior to the lipid extraction, which can significantly improve the lipid extraction yield by releasing intracellular lipid during the pretreatment (e.g. cell lysing). Hence, this work presents different pretreatment methods for improving lipid recovery from wet oleaginous microbes. The article is organized as follows, in the next section, a summary of different pretreatment techniques for lipid extraction is discussed. A detailed description of microbial cell disruption along with lipid extraction using both mechanical and non-mechanical technique is presented afterward. Then, a comprehensive discussion of microbial cell disruption and the effect of different factors on cell disruption using pressurized gas treatment is presented. After that, the energy consumption of some of the most commonly used methods for microbial cell disruption is discussed. Finally, a new pretreatment method is proposed to improve the lipid recovery from the wet route of microbial lipid extraction.

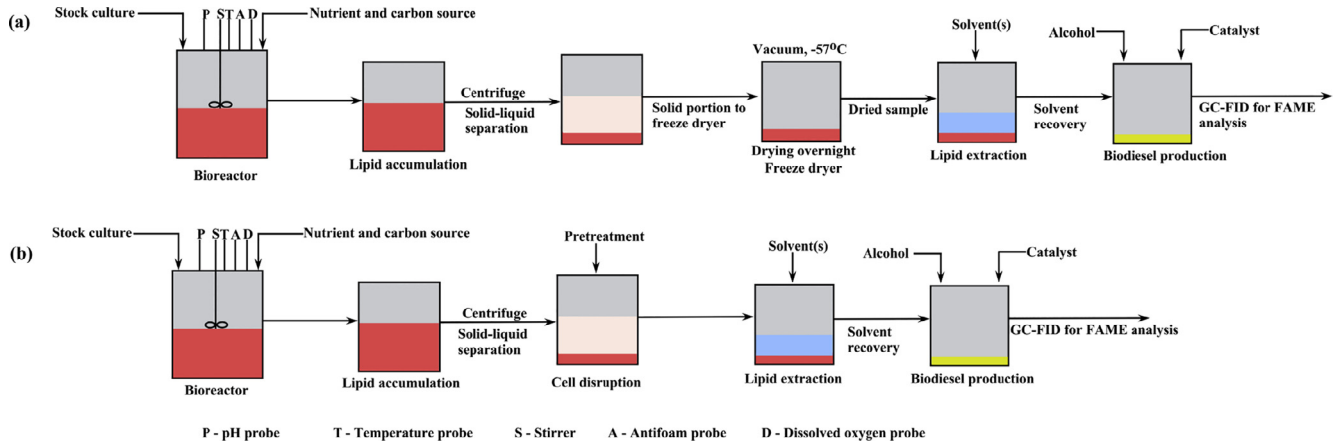

Fig. 1. Biodiesel production from oleaginous microorganisms using two different routes; a) biodiesel after drying the biomass, b) biodiesel directly from the wet biomass.

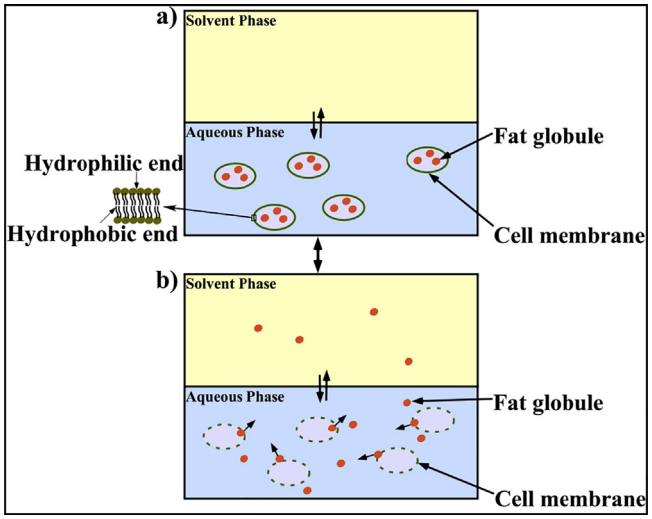

Fig. 2. Pretreatment of the wet microbial cell to release the intracellular lipid. a) Before the treatment, and b) after the treatment.

2. Different treatment methods for lipid extraction from wet oleaginous microbes

The presence of high moisture content (more than 80% on DCW) in the wet microbial cells inhibits the mass transfer of the solvent to the lipid layer as well as often leads to emulsion formation resulting in reduced lipid extraction efficiency (Dong et al., 2016a,b). Fig. 2 shows two forms of mass transfer limitations that need to be overcome to extract the lipid from wet biomass. The first is the interfacial barrier between the solvent and aqueous phase, which can be overcome by agitation, and the second limitation is the cell membrane that contains accumulated lipid in the microorganisms, which can be overcome by pretreating the cell prior to the solvent addition. After pretreatment, the intracellular lipid is released into the aqueous media. Subsequently, the solvent is added to the system to extract the lipid from the aqueous phase. Hence, a pretreatment is needed in wet biomass prior to the lipid extraction to improve extraction efficiency.

There are many pretreatment methods currently available for lipid extraction from wet oleaginous microbes, which can be broadly divided into two main methods: mechanical and non-mechanical (Middelberg, 1995). The mechanical method includes high-pressure homogenization, bead milling, ultrasound sonication, pulsed electric field, microwave assisted pretreatment, etc. The non-mechanical method is sub-categorized into three groups: physical, chemical, and enzymatic pretreatment. Physical pretreatment includes decompression, osmotic shock, and thermolysis; the chemical method includes antibiotics, chelating agents, chatropes, detergents, and use of different solvents. Lytic enzymes, autolysis, and cloned-phage lysis are currently used enzymatic assisted pretreatment techniques for microbial cell disruption. The mechanical method has been used extensively in comparison to other techniques due to their easier operation and scalability. Bead milling is a mechanical approach that creates a direct mechanical damage to the cell through the spinning of the beads placed inside a cylindrical compartment along with the treated cell. The bead milling technique works suitably well for a biomass concentration between 100 and 200 g/L (Show et al., 2015). Though cell disruption using bead milling has been shown to improve the lipid recovery from oleaginous microbes, the requirement of higher energy acts as a barrier for its large scale biofuel application (Lee et al., 2012a,b). Pulsed electric field (PEF) method uses the external electric field to create a critical potential in the cell resulting in cell disruption by creating a stress in the cellular membrane through the movement of charged particles. The lipid extraction using PEF can be increased up to 4.2-fold compared to the untreated cells (Luengo et al., 2015). There have been some limitations using PEF for cell disruptions such as the solution must be electrically non-conductive, needs sample prewashing, and cell disruption is highly dependent on medium composition (Gunerken et al., 2015). Cell disruption using osmotic shock occurs due to the addition of high concentration additives (e.g., salts) to the treated cell biomass resulting in sudden reduction of the movement of water molecules across the cell membrane. This method is very easy to operate e.g., the simple addition of additives, but needs longer operating time for the cell disruption (Show et al., 2015). A detailed discussion of some of the commonly used cell disruption techniques for microbial lipid extraction from wet microbes is presented in the following sections.

2.1. High pressure homogenization

High pressure homogenization (HPH) has been successfully applied in dairy industry to break up the fat globule in milk processing and in the agricultural industry to disrupt the unwanted microbes by applying high pressure (Geciova et al., 2002; Yusaf and Al-Juboori, 2014). The high pressure is usually achieved by passing the cell suspension through the smaller orifice of a discharge valve where the cell is pumped to the homogenizer using a positive displacement pump. The main parameters in HPH treatment are pressure (60–150 MPa), number of passes (1–10), and cell concentration (0.25–25% w/w CDW) (Drévillon et al., 2018; Jiménez Callejón et al., 2014; Yap et al., 2015). Recently, the HPH method has been applied to disrupt the microbial cell for improving the lipid recovery from wet oleaginous microbes. For example, Lucie et al. found 76% lipid recovery from wet biomass after HPH treatment for 5 passes at 150 MPa (Drévillon et al., 2018). Callejon et al. reported a 3-fold increase in lipid recovery from wet biomass containing 86% w/w water using hexane as the extraction solvent at low temperature (20–22 °C) (Jiménez Callejón et al., 2014). Though HPH has been a success in dairy or agricultural industry, this method has several drawbacks that require further investigation prior to its industrial-scale consideration for biofuels application. One of the major drawbacks for HPH is its suitability for high cell concentration only where the energy requirement is relatively lower compared to the low cell concentration. For low concentration, the energy requirement is very high making the process uneconomical due to the additional drying to reduce the excess water content (Gunerken et al., 2015; Yap et al., 2015). Also, the emulsion is formed during the HPH treatment making them difficult to separate the desired product in post cell disruption treatment (Dong et al., 2016a,b). Furthermore, the high energy requirement and maintenance of high pressure facility in HPH needs to be assessed carefully prior to the large scale applications for lipid recovery. For example, Lee et al. evaluated a comparative study on energy requirement for different mechanical methods and found that HPH treatment requires 529 MJ energy to treat 1 kg of dry biomass, which is higher compared to other mechanical methods (Lee et al., 2012a,b).

2.2. Microwave assisted pretreatment for lipid extraction

Microwave (MW) breaks the weak hydrogen bonds through enhanced rotation/vibration of solvent molecules (e.g., water); thus, increasing the solvent penetration to the cell interior resulting in complete cell wall disruption to release the intracellular lipid (Destandau et al., 2013; Lebovka et al., 2016; Trivedi et al., 2011). Extensive research has been conducted on microbial lipid extraction using MW assisted method, which is getting attention for improving biodiesel production from wet oleaginous microorganisms (Cheng et al., 2013; Cui and Liang, 2014; Hernández et al., 2014; Meullemiestre et al., 2016). The advantages of MW assisted method include the non-contact heat source, effective heating, faster energy transfer, improved production rate, reduced extraction time, and can utilize both dry and wet biomass for the treatment. For the pretreatment using MW assisted heating, the wet biomass is added into the sample reactor, which is

placed in a microwave system prior to the treatment. Distilled water can be added to the cell paste to increase the polarity as well as to prevent excessive heating during microwave exposure, which improves the efficiency of the microwave assisted method (Ali and Watson, 2015). The MW is set to a certain power (635–1400 W) and frequency usually at 2450 MHz to treat the dissolved wet biomass. The treatment is conducted for a certain exposure time (0–30 min) at a fixed temperature. After the treatment, the sample is cooled down to the room temperature and mixed with extraction solvent (hexane or chloroform) and co-solvent e.g., methanol in a certain ratio for the lipid extraction. After the extraction is finished, the solution is centrifuged to separate the lipid-containing solvent phase, and finally, the lipid is recovered gravimetrically by evaporating the solvent through running N_2 gas or using vacuum distillation (Balasubramanian et al., 2011). Sometimes in the MW assisted treatment, the extraction solvent(s) are added to the microwave facility prior to the treatment for direct lipid extraction. For that system, one important point needs to be considered is the treatment temperature (Khoomrung et al., 2013; Koberg et al., 2011). If the temperature is too high, solvent evaporation occurs, and the extraction efficiency is affected. To prevent solvent loss due to evaporation, a condenser is used to maintain a constant solvent volume during the treatment (Wahidin et al., 2014). On the other hand, if the treatment temperature is too low, the disruption efficiency is reduced. The efficiency of microwave assisted disruption and subsequent lipid extraction depends on the type of biomass. A thick cell wall containing microbes will require a long exposure time and higher temperature to get the complete disruption compared to the microbes with thin cell wall.

The MW irradiation method improves the overall lipid recovery from the wet biomass as reported in many publications (Ali and Watson, 2015; Balasubramanian et al., 2011; Wahidin et al., 2014). For example, Wahidin et al. recovered the total maximum lipid of 38.31 g/100 g CDW using the MW assisted pretreatment compared to 23.01 g/100 g CDW obtained from the conventional water bath system from an algae *Nannochloropsis* sp. (Wahidin et al., 2014). The effect of treatment temperature was studied by Balasubramanian et al. where they reported an increase in treatment temperature from 80 to 95 °C significantly improves the oil extraction from 24% to 33% on dry weight basis for an exposure time of 30 min (Balasubramanian et al., 2011). The lipid extraction efficiency can also be improved by changing the microwave power. For example, the lipid recovery of *N. oculata* increased from 0.036 g/g of wet algae to 0.052 g/g wet algae when the microwave power was increased from 635 to 1021 W (Ali and Watson, 2015). The microwave assisted lipid yield from wet microalgae *Nannochloropsis* sp. using ethanol as co-solvent was 65.4% compared to Soxhlet extraction (46.3%) that signifies the improvement of overall lipid content with the addition of a co-solvent (Saifuddin et al., 2016). The direct transesterification of the wet biomass can be applied to the microwave-assisted method that can save both time and improve extraction efficiency as reported in the literature (Chuck et al., 2014; Patil et al., 2013). Though direct transesterification can be used to produce biodiesel directly from the wet biomass, the yield is comparatively low (Cui and Liang, 2014). Sometimes, the overall lipid extracted from wet biomass using MW treatment is lower compared to the conventional methods because of lower solvent consumption and presence of moisture, which limits solvents mass transfer (Cheng et al., 2013). Though the extraction efficiency of MW pretreatment is usually high and suitable for laboratory-scale use, the high energy requirement poses a significant barrier for this method for its scalability (Lee et al., 2012a,b). Also, sometimes unstable bonds in the carbon chain structure is generated in MW treatment hampering the final products (Lee et al., 2017). Gude et al. reported that MW is inefficient for large-scale application due to its inability to penetrate through the large sample volume, which would impede its commercial application (Gude et al., 2013). Furthermore, the MW showed to be inefficient for cell disruption if the target compound is nonpolar and volatile (Zheng et al., 2011). Last but not least, the maintenance cost of MW system is also very high, which needs to be evaluated prior to its scale-up consideration.

2.3. Ultrasound assisted pretreatment

Ultrasound sonication (US) is a widely used mechanical method for microbial cell disruption, which uses high-frequency ultrasound to create a shear force for the cell disintegration (Liu et al., 2013). In the process, cavitation is associated with cell lysing which is the combination of formation, growth, and collapse of gas and vapor bubble (Gogate, 2011). In the collapse phase of cavitation, the pressure reaches thousands of atmosphere at the point of collapse when the sonic energy is converted to mechanical energy to increase the mass transfer of the solute in the solvent resulting in cell disruption (Liu et al., 2013). Though the US was mainly developed for food processing application (Cancela et al., 2016), it has been applied extensively to improve the lipid recovery either alone or with the combination of other pretreatments (Duarte et al., 2017). This method can be used to disrupt the cell wall followed by lipid extraction using a traditional solvent or it can be directly used for the transesterification of the cell's lipid to produce biodiesel. Similar to MW assisted pretreatment, the direct transesterification of treated cells using US is advantageous because it saves both the processing time and solvent consumption. For the pretreatment of wet oleaginous microbes using US assisted method, a defined quantity of wet cells is added to a sample tube that is placed in an ultrasound sonicator with a certain frequency (20–40 kHz) and power (1–1000 W) for a defined treatment time (5–60 min). After the treatment, a mixture of solvents (e.g., chloroform/methanol, hexane/methanol) or a single solvent (e.g., hexane) is added and the reaction continues for a certain time (Cheng et al., 2014). After completion of the process, hexane or chloroform is removed from the sample by distillation to determine the total lipid content gravimetrically. Similar to MW assisted method, solvent(s) can be added to the US system prior to the treatment for the direct extraction. The US frequency and power are tuned to find the optimum lipid recovery during ultrasound pretreatment, which is also dependent on the treatment temperature and processing time.

There have been a number of recent publications on improving lipid recovery from wet oleaginous microorganisms using the US assisted pretreatment. For example, Cheng et al. found a slight increase in the lipid content using the US compared to the Bligh and Dyer method (Cheng et al., 2014). Keris-Sen studied the effect of US on lipid extraction from wet microalgae using two solvent systems (with hexane and methanol/chloroform) where they found an increase in the lipid yield of 71% and 45% on dry weight basis for chloroform/methanol and hexane, respectively, compared to the untreated cell suspension (Keris-Sen et al., 2014). The US can be applied for the direct transesterification of the wet microbes, which is timesaving and requires lesser solvent consumption. For example, Martinez et al. conducted in situ transesterification of algae *Chlorella* sp. using US pretreatment with the addition of common solvents (e.g., ethanol) (Martinez-Guerra et al., 2014). Having many advantages to use in laboratory-scale, the US would create oxidative free radicals if the operation is continued for prolong period creating inhibitory effects to the product by reacting with intracellular biomolecules (Show et al., 2015). High heat generation is another concern that ought to be considered before large-scale application; otherwise, the overall process cost would increase due to the addition of cooling to maintain the desired temperature (Lee et al., 2017). The disruption efficiency using US also depends on the cell wall that microbe poses and the acoustic power the US applies during the operation. For example, Halim et al. found that US treatment was not able to disrupt the algal cell wall at 130 W for 25 min, which indicates higher acoustic power is needed for the cell disruption (Halim et al., 2012). Table 1 presents the comparison of lipid extraction yield using both MW and US with conventional methods.

Table 1
Microwave irradiation and ultrasound sonication assisted pretreatment for improving lipid recovery from wet oleaginous microorganisms.

Microorganism(s)	Treatment	Combination with other treatment	Optimum lipid extraction conditions	Lipid yield	Lipid increase	References
Chlorella PY-ZU1	MW	None	10 min, 80 °C, $CHCl_3$:CH_3OH-1:1	18.7 g/100 g dry biomass	0.92 times[b]	Cheng et al. (2013)
Yarrowia lypolytica	MW	None	1000 W, 30 min, 110 °C, $CHCl_3$:CH_3OH-1:2	7.13 g/100 g dry biomass	1.14 times[c]	Meullemiestre et al. (2016)
Nannochloropsis oculata	MW	None	1021 W, 5 min, H_2O + C_6H_{14} + C_2H_5OH	0.052 g/g dry biomass	3.25 times[d]	Ali and Watson (2015)
Scenedesmus obliquus	MW	None	95 °C, 30 min, C_6H_{14}	31.38 g/100 g dry biomass	1.64 times[d]	Balasubramanian et al. (2011)
Nannochloropsis sp.[a]	MW	None	180 W, 15 min, 40% biodiesel + 60% C_2H_5OH	5.23 g /8 g wet biomass	1.41 times[e]	Saifuddin et al. (2016)
Phaeodactylum tricornutum	MW	DES	150 °C, 30 min, DMC	12.5 g/100 g dry biomass	0.40 times[b]	Tommasi et al. (2017)
Pavlova lutheri[a]	US	None	300 W, 37 kHz, 30 min, $CHCl_3$:CH_3OH-1:2	47.2 g/100 g dry biomass	10.23 times[e]	Cancela et al. (2016)
C. pyrenoidosa	US	None	500 W, 30 min, $CHCl_3$:CH_3OH-1:1	11.3 g/100 g dry biomass	0.60 times[b]	Cheng et al. (2014)
Mixed culture	US	None	0.4Wml^{-1}, 30 kHz, 60 min, $CHCl_3$:CH_3OH-1:1	11.6 g/100 g dry biomass	1.71 times[c]	Keriş-Sen et al. (2014)
C. vulgaris	US	Homogenizer	750 W, 20 kHz, 60 min, $CHCl_3$:CH_3OH-1:1	23.75 g/100 g dry biomass	1.56 times[f]	Park et al. (2015)
Y. lypolytica	US	None	300 W, 30 min, 25 °C, $CHCl_3$:CH_3OH-1:2	8.1 g/100 g dry biomass	1.30 times[c]	Meullemiestre et al. (2016)

MW – Microwave pretreatment.
US – Ultrasound sonication.
DES – Deep eutectic solvents.
DMC – Dimethyl carbonate.
[a] Direct lipid extraction without pretreatment.
[b] Compared with Bligh and Dyer method (Extraction from dried biomass).
[c] Compared with Bligh and Dyer method (Extraction from wet biomass).
[d] Compared with control (no MW treatment).
[e] Compared with Soxhlet extraction.
[f] Compared with hexane extraction after US treatment.

2.4. Surfactant assisted pretreatment for lipid extraction

Surfactant-assisted cell disruption followed by lipid extraction can be a useful technique to improve the lipid recovery directly from the wet oleaginous microbes for biodiesel production. A number of recent studies have been conducted on lipid extraction by treating the wet biomass using different surfactants, which are nontoxic, sustainable, and biodegradable (Jeevan Kumar et al., 2017; Lai et al., 2017, 2016, Yellapu et al., 2017, 2016). Lai et al. reported up to 160-fold increase in the biodiesel feedstock production using isopropanol and hexane along with the pretreatment of Myristyltrimethylammonium bromide (MTAB)- and 3-(decyldimethylammonio)-propanesulfonate inner salt (3_DAPS) surfactants compared to the untreated biomass (Lai et al., 2016). Different types of surfactants (ionic, nonionic and zwitterionic) may be needed to disrupt the different types of microbial cell walls (Wu et al., 2011). During the process, the hydrophobic surfactant attaches to the hydrophobic cell membrane of the treated microbes and forms a micelle. When the critical micelle concentration is achieved, the micelle is separated from the cell membrane (Huang and Kim, 2013) and intracellular components (e.g., lipid) come out from the cell interior as a result of cell membrane breakage (Singh et al., 2007). The efficacy of cell disruption not only depends on critical micelle concentration but also on the properties of the cell membrane that microbes possess (Arachea et al., 2012; le Maire et al., 2000). Some of the microbes can easily be disrupted using the surfactant whereas others require longer exposure time to completely disrupt the cell wall due to the robust cell membrane. The use of a cationic surfactant with varying alkyl chain length (from C12 to C14) was studied by Lai et al. where they found that using C14 cationic surfactant up to 90% of the total lipid can be recovered using a nontoxic solvent ethyl acetate without changing the fatty acid profile from wet *Chlorella* microalgae (Lai et al., 2017). The surfactant-assisted method can be improved with the addition of another pretreatment method to increase the lipid recovery and reduce the extraction time. The combination of detergent N-Lauroyl sarcosine (N-LS) and ultrasound sonication decreased the extraction time from 12 h to 5 min for the direct transesterification of wet oleaginous yeast *Yarrowia lipolytica* (Yellapu et al., 2017). Though there has not been much research performed on lipid extraction from wet microbes using the surfactant assisted method, the cost of surfactants, the lipid composition in the cell culture, stage of the cultivation, and the microbe types are some of the constraints that need careful assessment for its scale-up consideration (Jeevan Kumar et al., 2017).

2.5. Enzymatic assisted method to improve lipid recovery

Enzymatic lysing is another cell disruption technique, which is capable of improving lipid recovery from the wet biomass by disrupting the rigid microbial cell wall using different enzymes. The enzymatic hydrolysis is advantageous because the process is carried out under mild reaction conditions that degrade a specific chemical linkage using specific enzymes. This method does not create any side reactions as in the case of chemical lysing. The enzymatic hydrolysis has been mainly applied for the lipid extraction from different oil sources such as soybean flour, sunflower seeds, and rice bran (Chabrand and Glatz, 2009; Sharma et al., 2001; Sineiro et al., 1998). The application of enzymatic hydrolysis on microbial cell wall disruption especially on microalgae was conducted by several researchers for biofuel application (Gerken et al., 2013; Harun and Danquah, 2011; Lee et al., 2013). The most common enzymes being utilized for microbial cell disruptions are cellulase, hemicellulase, lysozyme, papain, trypsin, neutral protease, etc. These enzymes can be used alone or as a mixture to find the difference in disruption efficiency. Generally, for enzymatic hydrolysis, the enzyme or the mixture of enzymes at a certain concentration (1–6%) is dissolved in the wet biomass. Before the enzymatic hydrolysis, the solution needs to be adjusted to a fixed pH (3.0–5.8) for the optimal enzymatic activity. The mixture is thoroughly mixed using a stirrer bar

Table 2
Microbial lipid extraction from oleaginous microbes by pretreating with different ionic liquids.

Microorganism(s)	Ionic Liquid(s)	Combination with another pretreatment	Optimum lipid extraction conditions	Lipid yield	Lipid increase	References
Nannochloropsis sp.[a]	[BMIM][Cl] [EMIM] [$MeSO_4$] [BMIM][CF_3SO_3]	MW	CH_3OH + [EMIM] [MeSO4], 15 min	36.79 g/100 g dry biomass[b]	1.28 times[c]	Wahidin et al. (2016)
Chlorella vulgaris	[BMIM] [$MeSO_4$]	US	C_6H_{14} + H_2O	47 mg/g DCW	1.62 times[d]	Kim et al. (2013)
C. vulgaris	[$P_{1\ 4\ 4\ 4}$][O_2CEt]	None	C_6H_{14} + CH_3OH	25 g/ 100 g dry biomass[b]	1.39 times[e]	Orr et al. (2016)
C. sorokiniana	[BMIM][HSO_4]	MW and US	Ionic liquid + H_2O	0.23 g/g dry biomass	15 times[d]	Pan et al. (2016)
N. salina				0.10 g/ g dry biomass	> 100 times	
Galdieria supphuraria				0.19 g/ g dry biomass	10 times	
Cyanobacteria[a]	[BMIM][Cl]	None	C_6H_{14} + CH_3OH	21.965 mg/g dry biomass[b]	0.97 times[f]	Kilulya et al. (2014)
Scenedesmus sp.	[$HNEt_3$][HSO_4] [BPy][HSO_4] [BPy][H_2PO_4]	Pressurized N_2	C_6H_{14}	35.7% (dry weight)	–	Yu et al. (2016)
				39.4% (dry weight)		
				23.5% (dry weight)		
C. vulgaris	[$P(CH_2OH)_4$][Cl]	None	[$P(CH_2OH)_4$][Cl] + CH_3OH	8.1 g/100 g dry biomass	1.17 times[d]	Olkiewicz et al. (2015)
N. oculata				12.8 g/100 g dry biomass	0.74 times[d]	
C. microalgae	[EMIM][[$MeSO_4$]	None	[EMIM][[$MeSO_4$] + CH_3OH, 18 h	38.0 g/100 g dry biomass	2 times[d]	Young et al. (2010)
C. vulgaris	[BMIM][CF_3SO_3]	None	[BMIM][[CF_3SO_3] + CH_3OH, 18 h	19.0 g/100 g dry biomass	1.72 times[d]	Kim et al. (2012)
C. vulgaris	[EMIM]OAc	Molten salts	[EMIM]OAc + $FeCl_3{\cdot}6H_2O$, 1 h	22.76 g/100 g dry biomass	1.04 times[g]	Choi et al. (2014)

[BMIM][Cl]: 1-butyl-3-methylimidazolium chloride; [EMIM] [$MeSO_4$]: 1-ethyl-3-methylimidazolium methyl sulfate.
[BMIM][CF_3SO_3]: 1-butyl-3-methylimidazolium trifluoromethane sulfonate; [BMIM] [MeSO4]: 1-butyl-3-methylimidazolium methyl sulfate.
[$P_{1\ 4\ 4\ 4}$][O_2CEt]: tributylmethylphosphonium propanoate; [BMIM][HSO_4]: 1-butyl-3-methylimidazolium hydrogen sulfate.
[EMIM]OAc: 1-ethyl-3-methylimidazolium acetate; [$P(CH_2OH)_4$][Cl]: tetrakis(hydroxymethyl) phosphonium chloride.
[$HNEt_3$][HSO_4] triethylammonium hydrogen sulfate; [BPy][HSO_4]: 1-butylpyridinium hydrogen sulphate.
[BPy][H_2PO_4]: 1-butylpyridinium dihydrogen phosphate.

[a] Direct transesterification.
[b] Biodiesel yield.
[c] Biodiesel was compared with conventional method (hexane and chloroform).
[d] Compared with Bligh and Dyer method (both ionic liquid, and B&D method extracted lipid from dried biomass).
[e] Compared with Folch method (both ionic liquid, and Folch method extracted lipid from dried biomass).
[f] Biodiesel was compared with conventional method (chloroform and methanol) using ultrasound sonication pretreatment.
[g] Compared with single ionic liquid pretreatment method.

during the dissolution for proper mixing.

After the incubation for a defined time (0–72 h), a common extraction solvent (e.g., hexane) is added to the mixture and then lipid is recovered by evaporating the solvent from the lipid-solvent mixture. The total lipid content is determined gravimetrically and extraction efficiency compared with untreated cells (Tommasi et al., 2017). Cho et al. reported an increase in the lipid extraction yield by 1.29–1.73-fold using cellulase enzymatic hydrolysis compared to the untreated cells (Cho et al., 2013). Hou et al. used the mixture of different enzymes for lipid recovery from wet green algae *Neochloris oleoabundans* where they found up to 86.1% lipid recovery using cellulase, pectinase, and hemicellulase in a ratio of 1:1:1 (Huo et al., 2015). Though enzymatic extraction has many advantages for improving the lipid recovery from oleaginous microbes, one of the main drawbacks for enzymatic oil extraction is the high cost of enzymes, which would make the process impractical to scale-up at the current state. The extraction efficiency is also lower in enzymatic extraction compared to solvent extraction (Puangsri et al., 2005). Further investigations are needed to consider the enzymatic extraction on commercial application. One way to improve the lipid recovery using enzymatic hydrolysis is to combine this method with other pretreatment techniques. For example, Wang et al. reported the improved lipid recovery of 92.6% using the combination of papain and cellulase enzymes with high-pressure homogenization (Wang et al., 2015). Jin et al. reported that the treatment of wet *Rhodosporidium toruloides* with recombinant β-1,3-glucomannanase (plMAN5C) improved the lipid recovery from 6.29% to 11.29% whereas the recovery increased to 62.2% when microwave irradiation was added prior to the enzymatic treatment (Jin et al., 2012).

2.6. Ionic liquid assisted pretreatment

Different ionic liquids can be used to pretreat both dry and wet microbial cells prior to solvent(s) addition in the lipid extraction process to improve the efficacy of lipid extraction process. There have been a number of recent studies on lipid extraction from different oleaginous microbes especially on microalgae using ionic liquid assisted pretreatment to improve the lipid extraction yield for biofuels applications (Bauer et al., 2017; Chen et al., 2015; Piemonte et al., 2016; Shankar et al., 2017; Yu et al., 2016). Ionic liquids are considered as green solvents and non-flammable due to their low vapor pressure. Some of the ionic liquids can form hydrogen bonds that can help dissolve the biomass so that the lipid from the intracellular region becomes available for extraction. For ionic liquid assisted treatment of the wet biomass, the biomass is mixed initially with the ionic liquid at the desired concentration and incubated for a fixed time for the proper dissolution of the ionic liquid in the biomass. The mixing of the solution is enhanced through continuous stirring during the dissolution process. The reaction is conducted in temperature ranges from ambient temperature up to 120 °C and the incubation time is approximately 10–60 min. The cell wall is disrupted due to the application of ionic liquid. High temperature and incubation time is desired for the complete lysing of the treated cells. After the treatment, the free lipid is floated in the cell suspension due to the nature of ionic liquids (insoluble in lipid), which can be extracted by centrifuging the solution and taking the upper phase (lipid). The process can be performed by adding water in the solution, and the ionic liquid can be recovered by evaporating the water from the solution after the treatment.

Different cell disruption techniques can be combined with ionic liquid assisted pretreatment to improve the lipid extraction considerably as reported in the literature (Kim et al., 2013; Pan et al., 2016; Wahidin et al., 2016). For example, Yu et al. treated *Chlorella vulgaris* using the combination of pressurized CO_2 with ionic liquids (1-butyl-3-methylimidazolium tetrafluoroborate [BMIM][BF_4] and 1-butyl-3-methylimidazolium chloride [BMIM][Cl]) where they found an increase in the lipid recovery from 68.0% to 75.6% when [BMIM][BF_4] was treated with pressurized CO_2 (Yu et al., 2015). The combination of ionic liquids with either microwave irradiation or ultrasound sonication have been studied by several researchers to improve the overall lipid extraction yield. For example, Kim et al. reported an increase in the lipid production from 47 mg/g to 75.2 mg/g DCW when ultrasound irradiation was combined with 1-butyl-3-methylimidazolium methyl sulfate [BMIM][$MeSO_4$] compared to the ionic liquid itself (Kim et al., 2013). Lipid extraction followed by a transesterification as well as the direct transesterification can be employed for biodiesel production from microorganisms using the ionic liquid assisted method. For example, Wahidin et al. used direct transesterification of microalgae using three ionic liquids (1-butyl-3-methylimidazolium trifluoromethane sulfonate [BMIM][CF_3SO_3], 1-ethyl-3-methylimidazolium methyl sulfate [EMIM][$MeSO_4$], and [BMIM][Cl]) (Wahidin et al., 2016). The pretreatment and the subsequent lipid extraction using different ionic liquids are presented in Table 2. One of the main advantages of ionic liquid assisted lipid extraction is the solvent free extraction process, which would be a promising approach due the environmental consideration. The high viscosity of ionic liquids sometimes hinders their lipid extraction efficiency, which can be overcome by combining this method with US pretreatment method (Kim et al., 2013). Though ionic liquid assisted extraction has a great potential, the knowledge of both cationic and anionic properties as well as the cost of bulk ionic liquids needs to be considered for its viability in industrial scale application (Jeevan Kumar et al., 2017).

2.7. Dilute acid pretreatment

Another promising cell disruption method for improving lipid recovery from wet oleaginous microorganisms is dilute acid pretreatment, which has been investigated recently by several researchers (Bai et al., 2014; Dong et al., 2016a,b; Lee et al., 2014). Different dilute acids have been investigated for their use in lipid recovery such as nitrous, sulfuric, and nitric acids. The main parameters in dilute acid treatments are acid concentration (0.5–10%), treatment time (15–30 min), and temperature (120–195 °C). It has been reported an improvement in the lipid recovery using dilute acid pretreatment method by Sathish and Sims where they recovered 79% of transferable lipid from wet algae containing 84% w/w moisture (Sathish and Sims, 2012). One important advantage of dilute acid treatment is the lower energy requirement compared to the mechanical method as reported by Dong et al. where they reported the energy requirement for dilute acid pretreatment is 2.5 MJ/kg compared to bead milling requiring 10.2–36.1 MJ/kg (Dong et al., 2016a,b). Though a significant quantity of cell disruption found at higher acid concentration (8%), temperature (160 °C), and treatment time (45 min), this process is not feasible for large scale application because the high acid concentration would be inhibitory to the desired product and the acid might be corrosive to the metallic reactor in industrial-scale application (Halim et al., 2012). Also, the disposal of water with low pH in post acid treatment would be a major concern because certain environmental regulations have to be followed prior to dispose the acid treated water. The dilute acid treated water with low pH needs to be neutralized prior to releasing to the environment. Furthermore, the recovery of dilute acid and its related cost after the treatment needs careful assessment prior to its scale-up consideration.

2.8. Pressurized gases for improving lipid recovery from wet microbes

Pressurized gases (e.g., CO_2, N_2, N_2O, and Ar) have traditionally been used for microbial cell disruption to inhibit the microbial cell growth in food preservation application, and there have been recently published works on microbial cell disruption using these gases to find their efficacy in cell inactivation (Garcia-Gonzalez et al., 2007; Liao et al., 2010). The use of different pressurized gases is advantageous compared to other methods because these gases (e.g., CO_2) are non-toxic, sources are cheap, easily available, and nonflammable. The desired microbial cell inactivation can be obtained using these pressurized

gases at different optimum conditions for different microbes. For example, Fraser used pressurized gas to rupture the bacterial cell wall and obtained 90% cell lysing under optimum conditions (FRASER, 1951). Different pressurized gases can be used to find their efficacy on cell disruption. For example, Enomoto et al. reported the cell viability under the treatment of CO_2, NO_2, N_2, and Ar using the same treatment condition (4000 kPa, 313.3 K, and 240 min) and found a log reduction of 6.8, 4.7, 0.03, and 0.01, respectively, which showed CO_2 effected 99% cell death compared to Ar and N_2 where only 1% cell death occurred (Enomoto et al., 1997). Nakamura et al. treated *Saccharomyces cerevisiae* yeast cells with pressurized CO_2 and N_2 at different exposure time. They found the survival ratio of the wet cells were reduced to $1/10^8$ when treated with CO_2 at a pressure and temperature of 4000 kPa and 313 K, respectively, for an exposure time of 3 h. They also found that CO_2 was superior to N_2 in terms of disruption efficiency because when treating with N_2, they found more than 92% of cells were survived (Nakamura et al., 1994). Although N_2O has nearly the same efficiency, CO_2 was found to be the most suited gas to treat the microbial cell because of its physical properties as well as the reactivity with the cell suspension during the pressurization.

For microbial cell disruption using pressurized CO_2, the wet cell suspension is placed in a high-pressure vessel prior to the start of the treatment. The system is then pressurized with CO_2 at the desired pressure [1000–4000 kPa] and temperature [0–40 °C] for a certain exposure time (Nakamura et al., 1994). A stirrer is used during the pressurization to enhance the gas–liquid mass transfer to improve the disruption efficiency of the treated gas. The system is suddenly depressurized (the pressure is released) at the end of the treatment as quickly as possible (approximately 100 kPa/s) and the sample is taken for analysis. Since pressurized CO_2 has been successfully applied to disrupt the microbial cell wall in the food industry, this technique can also be applied as a potential pretreatment method for improving lipid recovery from wet oleaginous microbes. From preliminary results, lipid recovery can be improved after treating the wet biomass with pressurized CO_2 as reported by Howlader et al. where they found up to 40% increase in the lipid recovery from an oleaginous yeast *Rhodotorula glutinis* when treated with pressurized CO_2 at 3500 kPa compared to the untreated cell (Howlader et al., 2017a). They reported a complete cell death (from plate counting) to obtain a 40% increase in the lipid recovery (0.352 g/g CDW from treated biomass compared to 0.251 g/g CDW from control). Cell viability using plating is certainly a useful technique to quantify the cell disruption, but the cell can die for many reasons and may not lead to improvement in lipid recovery. This claim is supported by the finding of Pagan and Mackey where they treated different strains of *Escherichia coli* bacteria applying high hydrostatic pressure and found more than 99% cell death, but the cell membrane was resealed after the decompression (Pagán and Mackey, 2000). Generally, the intracellular components are liberated from the cell due to the breakage of the cell membrane, which is generally characterized using scanning electron microscopy. In their article, Howlader et al. also visualized a morphological change in the cell wall in case of complete cell death where they found that the cell outer structure was completely damaged when treated with CO_2 (3500 kPa) compared to the untreated cell (the cell surface looked intact and no damage was observed). In summary, cell disruption using pressurized CO_2 is a promising technique that can be used to improve the lipid recovery from wet microbes, but only some preliminary results are available in the current state. Further research is needed to understand this process before assessing for industrial-scale consideration. Similar to cell disruption using pressurized CO_2, steam explosion is another approach currently understudy to improve the lipid recovery from oleaginous microbes (Al Hattab and Ghaly, 2015; Lorente et al., 2015). For example, Lorente et al. found a higher lipid content from microalgae when treated with 0–10% sulfuric acid (Lorente et al., 2015). Though steam explosion in combination with dilute acid treatment can be highly effective cell disruption technique to release intracellular lipid, the cost for producing steam would pose a strong barrier to its large-scale consideration. Since pressurized CO_2 was found to be suitable for cell disruption at low temperature and pressures [from 0 to 40 °C, and 1000–4000 kPa] for improving the lipid recovery from wet biomass, this method can have favorable energy input when compared to the steam explosion. The effect of different factors, the role of solubility, and mechanisms of cell disruption using pressurized CO_2 are discussed in the subsequent sections to get an overview of the pressurized CO_2 treatment.

2.8.1. Pressurized CO_2 for cell disruption: Effect of different factors, role of CO_2 solubility, and mechanism of the process

The main factors that affect the microbial cell disruption using pressurized CO_2 are pressure, temperature, exposure time, agitation, water content, depressurization rate, pressure cycling, type of microorganisms, etc. (Garcia-Gonzalez et al., 2007; Melo Silva et al., 2013; Soares et al., 2013). One of the most important factors in microbial cell disruption is the applied pressure. Generally, the solubilization of CO_2 increases with the increase of pressure because the solubility of CO_2 in water, grown cell suspension, spent media, and lipid increases as the pressure increases (Howlader et al., 2017a, 2017b; Lucile et al., 2012). Exposure time is another important factor that signifies the effectiveness of microbial cell disruption. The exposure time can be varied from 1 min to several hours depending on the type of microbes and other parameters (da Silva et al., 2016; Garcia-Gonzalez et al., 2010; Nakamura et al., 1994). When the microbes are exposed to the pressurized CO_2 for a certain exposure time, some of the gaseous molecules penetrate through the cell wall, and as a result, the intracellular components come out from the cell. Generally, a longer exposure time is effective to breakdown the cell wall to obtain the desired products. The cell lysing can be done either subcritical or supercritical pressurized conditions. It would be economically advantageous if the experiment can be conducted using subcritical CO_2 due to low energy requirement for achieving the subcritical thermodynamic state. Though the use of expensive supercritical CO_2 can improve the cell inactivation, it would not be feasible due to the high processing cost. Farukawa et al. reported that the desired cell disruption can be obtained using the subcritical CO_2 where they found that the change of physical state of CO_2 from subcritical to supercritical state did not improve the cell inactivation sharply for different bacteria (Furukawa et al., 2009). The other factors that affect the cell disruption are depressurization rate, pressure cycling, water content, the susceptibility of other microbes in the cell suspension, agitation, and physical and chemical properties of the suspending medium (Garcia-Gonzalez et al., 2007).

The solubility of CO_2 in lipid as well as in lipid-rich microbial cell is also important because during the pressurization step the non-polar CO_2 interacts with the hydrophobic part of the lipid and diffuse through the phospholipid cell membrane. The solubility of CO_2 in lipid-rich microbial cell suspension is reported by Howlader et al. where they found that the solubility of CO_2 in the lipid-rich microbial cell was slightly lower than the solubility of CO_2 in water, but higher than the CO_2 in growth media and spent media (Howlader et al., 2017a). The presence of lipid in the grown cell culture increases the solubility due to the nonpolar interactions of CO_2 and lipid, but the presence of insoluble carbohydrates and protein in the cell suspension decreases the overall solubility. Hence, the solubility of CO_2 in lipid-rich cell culture was slightly lower than that of CO_2 in pure water. The solubility of CO_2 in lipid (triglyceride) was found to be considerably higher compared to the CO_2 in water (Howlader et al., 2017b). The structural and chemical properties of triglycerides and CO_2 are the reasons for the higher solubility. A recent report on the solubility of CO_2 in triglyceride indicated that carbon tail of the CO_2 interacts well with the carbonyl carbon and carbonyl oxygen of the triglycerides, which increases the CO_2 solubility in triglycerides (lipid) (Howlader et al., 2018). Since the solubility of CO_2 in lipid-rich cell suspension is higher compared to the CO_2 in both sugar broth medium and spent medium, and CO_2 is highly

soluble in lipid, the microbial cell disruption can be improved using the pressurized CO_2.

Though there is no defined mechanism for microbial cell disruption using pressurized CO_2 available to date, the process starts with the solubilization of CO_2 in lipid-rich microbial cells. Depending on the applied pressure and temperature, some of the CO_2 molecules solubilized in the cell suspension and pass through the gas–liquid interface to the liquid phase (aqueous media). CO_2 reacts with the water present in the cell suspension and reduces the external and internal *pH* of the media (Garcia-Gonzalez et al., 2007). It has been reported that the cell's activity is optimum at a certain *pH*, and the cell activity decreases with either decrease or increase in the *pH* because key enzymes in the cellular metabolisms are deactivated due to the sudden pH change (Garcia-Gonzalez et al., 2007). Some of the unreacted carbon dioxide molecules contact with the cell membrane and pass through the cell interior (Isenschmid et al., 1995). In the cell interior, CO_2 reacts with some of the vital components such as Mg^{2+} and Ca^{2+} of cells to precipitate as $CaCO_3$ and $MgCO_3$ (Garcia-Gonzalez et al., 2007). Due to the release of certain important metabolites from the cell cytoplasm, cells lose their normal activity. Due to these facts, the cell wall of the microbes is disintegrated and important components from the cell interior come out in the liquid phase. The main components that come out from the cells are protein, lipid, and carbohydrate that can be extracted using different solvents.

3. Different pretreatment methods from the energy consumption perspective

It is imperative to have the data on energy consumption of each pretreatment method to find the most cost effective and reliable way to extract the lipid from wet biomass. The energy consumption for high pressure homogenization (HPH) can be obtained by multiplying the applied pressure (MPa) with the amount of wet cell suspension processed (m^3), which is generally expressed as MJ/kg of dried biomass. On the other hand, the energy consumption for microwave irradiation, ultrasound sonication, and bead beating can be determined with knowledge of the power ratings, treatment time, and amount of wet cell or dried biomass. The energy consumption (MJ/kg of dried biomass) for cell disruption of some of the most common methods are presented in Table 3, where the lipid recovery and optimized cell disruption conditions are also provided. It can be seen from the table that the energy consumption for cell disruption using different methods varied from 2.19 to 891 MJ/kg dried biomass. For example, the energy consumption for HPH varies from 2.19 to 540 MJ/kg, which has already been used in industrial application. It is interesting to note that the energy consumption at low solid concentration (0.1% w/w) is significantly higher for HPH compared to the energy consumption at high solid concentration (25% w/w) for the same lipid recovery, which supports that HPH would be suitable for industrial scale application for lipid production (Yap et al., 2015). To make the process feasible, additional energy will be needed to make the biomass concentration from 0.1% (w/w) to 25% (w/w) prior to HPH treatment which should be taken into account for the scale up considerations. The bead beating treatment would not be feasible in large scale application due to its high energy consumption of 504 MJ/kg of dried biomass (Lee et al., 2012a,b). The energy consumption for microwave irradiation and ultrasound sonication varies from 6.0 to 890.0 MJ/kg of dried biomass, which is higher compared to the HPH method and has been used only in laboratory scale till now. An estimate of energy consumption of pressurized CO_2 was also determined using the operating conditions namely pressure, treatment time, and agitation, which is also provided in the table. There are three forms of energy consumption for pressurized CO_2 treatment: one for the applied pressure, one for the temperature rise from the room temperature (25 °C) to the treatment temperature, and one for the agitation of the sample at the desired exposure time. The applied pressure for the pressurized CO_2 treatment was 3500 kPa to

Table 3
Energy consumption for different pretreatment methods for microbial cell disruption.

Treatment Method	Microorganism	Optimized conditions	Lipid yield	Energy consumption (MJ/kg CDW[a])	References
High pressure homogenization	*Chlorococcum* sp.	200 mL, 1.7 g dry cell, 2500 W, 6 min		529.4	Halim et al. (2012), Lee et al. (2012a,b)
	Nannochloropsis sp.	0.1% w/w CDW, 150 MPa	30 g/100 g CDW	540.2	Yap et al. (2015)
	Nannochloropsis sp.	25% w/w CDW, 150 MPa	30 g/100 g CDW	2.2	Yap et al. (2015)
Bead Beating	*Botryococcus* sp.	100 mL, 0.5 g dry cell, 840 W, 5 min	28.10 g/100 g CDW	504	Lee et al. (2012a,b), Lee et al. (2010)
Microwave	*N. oculata*	4.3 g wet cell, 1 g dry cell, 1021 W, 5 min	5.2 g/100 g CDW	306.2[b]	Ali and Watson (2015)
	Yarrowia lypolytica	10 g wet cell, 1 g dry cell, 20 W, 15 min	7.13 g/100 g CDW	18.0	Meullemiestre et al. (2016)
	Scenedesmus sp.[c]	2 g dry cell, 1000 W, 10 min	29.65 g/100 g CDW	300.0	Guldhe et al. (2014)
	Botryococcus sp.	100 mL, 0.5 g dry cell, 700 W, 5 min	28.60 g/100 g CDW	420.0	Lee et al. (2012a,b), Lee et al. (2010)
Ultrasound sonication	*Chlorella pyrenoidosa*	10 mL cell suspension, 1.01 g dry cell, 500 W, 30 min	11.3 g/100 g CDW	891.0	Cheng et al. (2014)
	Scenedesmus sp.[c]	2 g dry cell, 100 W, 2 min	19.85 g/100 g CDW	6.0	Guldhe et al. (2014)
	N. oculata[d]	100 g wet cell, 5% w/w CDW, 1000 W, 30 min	0.48%[e]	3.6	Adam et al. (2012)
Pressurized CO_2	*Rhodotorula glutinis*	250 g cell suspension, 2.5 g dry cell, 3500 kPa, 5 h,	35.2 g/100 g CDW	232.6	Howlader et al. (2017a)

[a] CDW-Cell dry weight.
[b] Excluding the effect of volume fraction.
[c] Simultaneous cell disruption and lipid extraction.
[d] Solvent free extraction.
[e] The oil extraction at 0.48% (12 times lower lipid recovery) compared to at 5.76% for Bligh and Dyer method.

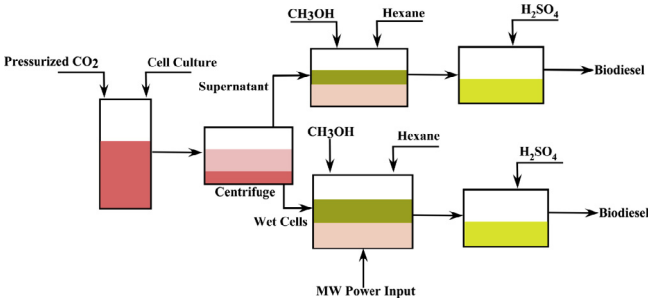

Fig. 3. Proposed biodiesel production from wet biomass using the combination of pressurized CO_2 and microwave irradiation.

treat 250 g of cell suspension (2.5 g dried mass), which corresponds to 0.35 MJ/kg dried mass. The energy consumption for the temperature increase from 25 to 30 °C was 2.09 MJ/kg dried biomass. Finally, the energy consumption for stirring the cell suspension using pressurized CO_2 at 300 rpm for 5 h was 230.11 MJ/kg of dried biomass. The total energy consumption for pressurized CO_2 treatment was 232.55 MJ/kg dried biomass having 1.006% (w/w) solid content, which is comparable to the HPH treatment, which needs high energy at low solid concentration. As the majority of the energy cost is associated with stirring, it can be reduced significantly by proper designing of the stirring process. The pressurized CO2 is a promising method, which can be combined with other methods to improve the lipid extraction efficiency and to reduce the energy consumption.

4. Future direction on microbial lipid recovery from wet biomass using pressurized CO_2

Microbial cell disruption using different pretreatments can be a useful approach to improve the lipid extraction directly from the wet biomass without energy-intensive drying for large-scale biodiesel production. The use of pressurized CO_2 for microbial cell disruption is advantageous compared to other methods because a complete cell lysing is obtained utilizing the CO_2 treatment in a reasonable time. This method can help to improve the lipid extraction by releasing the desired intracellular compounds such as lipid in particular, which is a very important metabolite obtained from oleaginous microorganisms. Although pressurized CO_2 is a promising technique, further research is needed to fully understand the process and the economics for large-scale applications. One of the possible potential methods for improving lipid recovery using the pressurized gas treatment is to combine this technique with other pretreatments to improve the disruption efficiency as well as lipid recovery from wet biomass. The use of a combination of different pretreatment methods have already been applied and improvement in the overall lipid content is reported compared to a single pretreatment method. The pressurized gas treatment followed by microwave (MW) irradiation or ultrasound sonication (US) can be a combined approach to improve the overall lipid recovery because these methods are being studied extensively and are promising when combined with other treatments. The addition of MW or US pretreatment is suggested because several comparative methods have been reported using different pretreatment methods for improving lipid recovery from wet biomass, and these two methods are found to be the optimal treatment due to the higher lipid recovery as well as other advantages such as lower processing time, lower solvent requirement, and easy application (Lee et al., 2010; Martinez-Guerra et al., 2018; Prabakaran and Ravindran, 2011; Zheng et al., 2011). Also, one study showed that the MW treatment alone has a very poor lipid yield, but the lipid content increased dramatically when the MW is combined with another pretreatment (Jin et al., 2012). The proposed cell disruption and subsequent lipid extraction from wet biomass could be as follows, the grown cell suspension after harvesting from the culture will be directly placed in a high-pressure reactor where a pressurized gas treatment (e.g., CO_2) will be applied to initially disrupt the cell wall of the microbes. The optimized cell disruptions conditions will be utilized to perform the pressurized CO_2 treatment. In the second step, the pressurized gas treated cell suspension will be centrifuged and the wet cell pellets will be taken to the MW or US treatment facility to extract the lipid using hexane as the solvent. The supernatant (liquid solution after the centrifuge) will also be used for lipid extraction because it was found that the liquid portion contains a considerable amount of lipid after the pressurized gas treatment (Howlader et al., 2017a). Finally, the fatty acid compositions of biodiesel obtained from both solid cell pellets and supernatant will be compared to meet the ASTM D6751 or EN 14214 standard. From the earlier results, the fatty acid composition of both the treated and control cell suspension was found to be unchanged upon the pressurized gas treatment (Howlader et al., 2017a). Fig. 3 demonstrates the proposed method on lipid recovery using pressurized CO_2 pretreatment followed by MW-assisted lipid extraction.

5. Conclusion

This work presents the detailed review of the microbial cell disruption using different techniques i.e. microwave irradiation, ultrasound sonication, use of ionic liquid, detergent, and different enzymes for improving the lipid recovery from wet oleaginous microorganisms. A new method named pressurized gas (CO_2) treatment for cell disruption has been presented, which was previously used for microbial inactivation for food preservation applications. Different factors affecting cell disruption and mechanism of pressurized gas treatment have been demonstrated. Finally, a new cell disruption method is proposed using the pressurized CO_2 treatment followed by the microwave-assisted method to improve the overall lipid extraction yield.

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