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First developments towards closing the nutrient cycle in a biofuel production process

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ABSTRACT

Hydrothermal liquefaction (HTL) of microalgae is a promising technology offering production of biofuels in a sustainable way. Thanks to the supercritical conditions applied, the recycling of water and nutrients including carbon capture becomes feasible[1–2]. Through HTL, the target bio-oil is generated together with process water containing valuable nutrients. In this study we have provided an environmentally friendly and resource efficient strategy to produce renewable microalgae biomass. Feasibility test on microalgae cultivation, using the nutrient-rich effluent from the HTL of same algal biomass, was performed in a 5 L, flat panel airlift (FPA)–photobioreactor (PBR). 1 g(DW) L^{-1} d^{-1} of microalgae was the productivity average achieved with the diluted aqueous solution (25-fold). This is comparable to the productivity obtained with the standard growth medium even if an adaptation time of four days was necessary. Our results show that the nutrient rich HTL aqueous product of microalgal biomass conversion can replace to a great extent mineral salts of the culture medium. Together with an efficient water management, the nutrient recovery applied here promotes the recycling and prevents the release of waste materials and therefore shows the potential for driving the system towards a nutrient neutral production of biomass for biofuels.

1. Introduction

Biofuels are considered low-carbon fuels and the best option for substituting the limited fossil fuels and mitigate the CO_2 emissions [3]. However, the existing technologies pay little attention to a) energy and eco-efficiency of possible fuel production pathways, b) the loss of valuable nutrients, such as phosphorus which is itself a resource limited material, c) the water management, and d) resulting fuel costs. Limiting the consumption of critical resources such as agricultural land, fertilizers and water, is the best way to efficiently reduce the greenhouse gas emissions.

Microalgae have been suggested as an advantageous energy feedstock for the production of biofuels because its mass production is possible without the need of valuable soils and because their production efficiency per area is higher than for plants [4,5]. However, most of the conversion pathways of microalgal biomass are energy intensive processes (due to pretreatment e.g. dewatering, extraction, fuel generation, etc.) and the efficiencies achieved were still low [6]. In addition, huge quantities of nutrients are involved in microalgae cultivation compromising the sustainability of the process not only with respect to mineral resources but also to the environmental and economic impacts because their production is highly energy intensive, a point very often neglected [7]. A study [8] has shown that if all of the transport fuel would be replaced by microalgae fuel in the EU, it would need around 25 million tons of nitrogen and 4 million tons of phosphorus per year. Therefore, approaches aiming at reducing the amount of fertilizers and improving in general the economic viability and the environmental sustainability of biofuels from microalgae should be implemented. One way already explored is to obtain the nutrients from renewable sources like wastewater or anaerobic digestion effluents [9–11]. However, important additional infrastructure would be required to deliver the waste streams to the algae production facilities and may increase the risk of contamination of the algae cultures by bacteria or other unwanted microorganisms.

In this context, what could be a convenient technology of conversion for microalgae? Hydrothermal conversion of biomass into fuels is an ecologically sound process as water is used as a "green" reactant and solvent instead of dangerous organic solvents. Additionally, no biomass drying is necessary due to the hydrothermal environment which is particularly suited for converting wet biomass into fuels with a high heating value. Finally, the thermal efficiency that can be achieved, defined as the energy in dry biomass to the energy in fuel produced, is

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Table 1
P. tricornutum culture conditions.

	Cultivation parameters
Medium	Mann & Myers with $(NH_4)_2CO_3$ [25]
Temperature	21 °C
pH	7.3–7.5
Airflow	200 L/h
CO_2	2.5–3.5% (v/v)
Light intensity	100–400 μE m^{-2} s^{-1}

70% with the hydrothermal pathway which is a factor 2 higher than reported fermentation processes efficiency [12]. Hydrothermal processes are usually classified according to their final product. Thus, in hydrothermal liquefaction (HTL) the main product obtained is bio-oil while in supercritical water gasification (SCWG) a valuable flue gas is produced. In some cases, HTL can be used as a pretreatment for SCWG.

Several research groups are currently working towards demonstrating the technical and economic feasibility of synthetic natural gas production via hydrothermal processing of microalgae [1,2,13–16]. A major innovation coming from one of these works regards a crucial process step where salts contained in the biomass are precipitated to a high extent (due to their low solubility in supercritical water) inside a salt separator prior to the biomass slurry reaches the catalytic hydrothermal gasification unit. As proposed by Stucki et al. (2009) [1] and Haiduc et al. (2009) [2], the salts may be used as fertilizers for growing microalgae in a PBR. First sensitivity analysis of this process known as "SunCHem" showed a 56% energy efficiency for self-sufficient systems where raceway ponds were used and the electricity needed (for microalgae cultivation and to run the hydrothermal plant) was provided by conversion of part of the syngas [17]. Such an efficient algae-to-methane process opens up a smart way to tackle both climate change and dependence on fossil natural gas without competing with food production.

Very few studies addressed the potential of nutrient recycling from the hydrothermal pathway. Tsukahara et al. (2001) [13] demonstrated the feasibility to recover nutrients from *Chlorella vulgaris* by low temperature gasification of the microalgae. Jena et al. (2011) [18] have confirmed that it is possible to cultivate microalgae in the process water following HTL of the freshwater microalgae but they achieved only a 50% biomass density when compared to the standard growth medium. Biller et al. (2012) [19] performed microalgae growth trials in the recycled process water using 4 strains but algae grew slower and reached a lower final concentration on the diluted process water than on standard media. Finally, Zhou et al. (2013) [20] used an algae-bacteria culture to test the effect on growth of a mixture of filtered primary municipal wastewater effluent and the recycled diluted HTL aqueous phase. Even if the precedent studies opened and strengthened the way for nutrient recycling, none of them demonstrated their applicability in efficient biomass production systems like PBRs. This type of cultivation system despite requiring expensive investment costs, ensures a high photosynthesis rate even at high cell concentrations and their energy requirements for algae production are much lower (556.9 TJ a^{-1}) than the energy content of the algae biomass produced (1985 TJ a^{-1}) [17,21].

In a key preliminary experiment, biomass of the microalgae *Phaeodactylum tricornutum* (20 wt.% DW) grown in a semicontinuous system was hydrothermally converted into oil and the recovered nutrient-rich aqueous effluent was sent back to the algae growth system to be used as culture medium. The main purpose was to evaluate if the nutrients obtained in the hydrothermal process were accessible by the algae and if the remaining organics did not show any toxification effects. *P. tricornutum* was chosen due to its high growth rate, high content of eicosapentaenoic acid and the reported highest volumetric productivity [22] (see Table 2 for its chemical composition). In addition, previous tests with *P. tricornutum* were successful in demonstrating that these algae can grow well in wastewater streams from anaerobic digestion biogas plants [23]. Therefore, this strain should be less sensitive than others to possible inhibitors on the HTL aqueous effluent and showed already great potential for mass cultivation in FPA bioreactors [21,24].

2. Materials and methods

An algae slurry of 20 wt.% DW (ca. 14 g *P. tricornutum* UTEX 640 + 56 g Water) was hydrothermally reacted in a batch using an autoclave 500 mL (Premex®) at a heating rate of 5 °C/min until 350 °C was

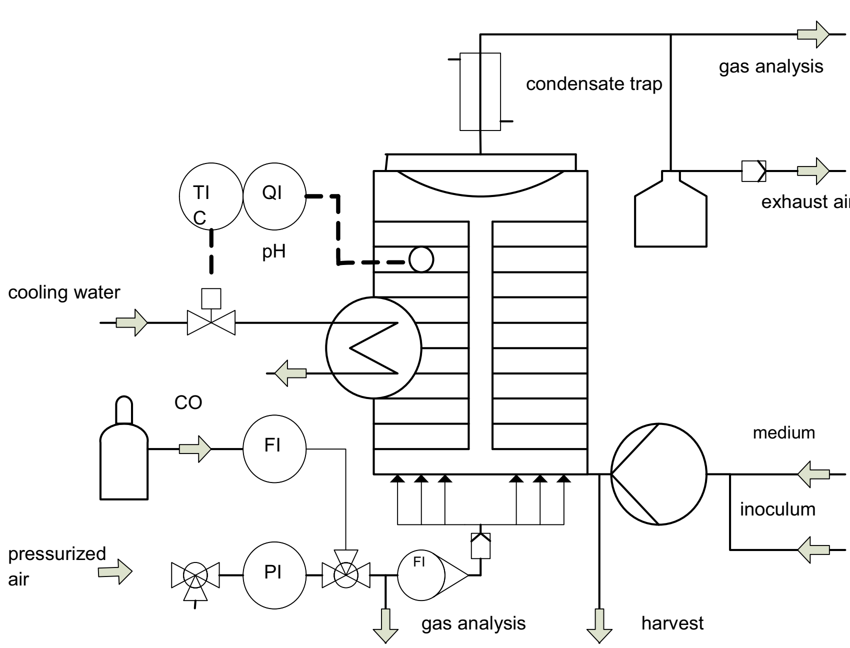

Fig. 1. Schematic diagram of the FPA-PBR system. Major components in the reactor include a temperature and pH-controller, a temperature-controlled water bath and two rotameters for the airflow and the CO_2 control. TI: temperature indication, QI: quality indication (pH), FI: flow indication and PI: pressure indication.