



Application of aqueous alkaline extraction to remove ash from algae harvested from an algal turf scrubber



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ABSTRACT

Efforts to develop algae as a sustainable feedstock for the bioenergy economy have largely focused on cultivating microalgae for optimized lipid production. Because of this, filamentous algae are often overlooked in the algae biomass field because of their relatively low lipid content, although they do contain high levels convertible carbohydrates and proteins. Systems designed to produce filamentous biomass, such as algal turf scrubbers (ATS), have advantages over planktonic algae produced in raceway systems. Unlike raceway algae production, ATS systems do not require inoculum, nutrients, or CO₂ inputs as these are readily obtained from the atmosphere or the natural waters feeding them. ATS systems are exceptional at removing nutrients from affected waterways. However, as a consequence of the growth environment (sand, silt and other suspended solids), and the composition of ATS biomass (diatoms), the periphytic biomass obtained from ATS can have very high ash, which represents non-convertible material that can complicate downstream conversion processes. We explored both physical and chemical approaches to remove ash with the goal of improving overall preprocessing costs and conversion yields while minimizing the loss of organic material. The simplest method for removing ash involved repeatedly rinsing the biomass with water at 25 °C, which removed 34.5 ± 3.4 wt% of the ash with no appreciable loss in biomass. When treated with 2.0 wt% NaOH at 80 °C, up to 87.8 ± 1.4 wt% of ash was removed. These severe conditions, however, also resulted in organic material losses of 29.9 ± 3.2 wt%. Ultimately, these results will inform future tests, both chemical and mechanical, and will provide input for models that identify energy bottlenecks and potential savings.

1. Introduction

Algae harvested from algal turf scrubbers (ATS) is one potential biomass material that can be economically produced, applied for a primary purpose (water remediation), and then potentially redirected as a conversion feedstock for bioenergy use. Algal turf, a community of naturally occurring periphyton, which self-attach to a substrate in the ATS flow way, grow mixo- and photoautotrophically, utilizing solar energy, nitrogen, phosphorus, trace metals, dissolved CO₂ and bicarbonate, and other naturally occurring carbon sources present in the source waters. Algae turf produced in ATS systems, unlike algae biomass produced in open raceway ponds, does not require inoculum, nutrients, or CO₂ inputs as these are readily obtained from the natural waters feeding them. In fact, a critical component of ATS systems is integration with contaminated water systems laden with excess nitrogen and phosphorus (such as brines, brackish eco-systems,

agricultural run-off [1], or even landscaping applications, such as aquariums [2]), preventing algal blooms and the ecological and economic damage they cause. Integration of ATS systems with contaminated water sources reduces the major nutrient inputs required to maintain high growth rate production while providing an environmental service, and recent analyses suggest that increased incorporation of ATS systems can significantly increase agricultural nutrient recycling and reuse on a global scale [3]. The fact that ATS systems are based on a native culture that dynamically adapts to changing conditions decreases culture crash events commonly seen in monocultures [4,5] and yields biomass with characteristics that make it easily harvestable using commonly available farm equipment [6]. ATS systems have been used commercially for contaminated water treatment with the produced biomass representing a potential co-product to water reclamation [7,8]. The stability of the systems and promising productivities make ATS a system of interest as a biomass production platform for

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biofuels. However, while cultivation of native periphyton improves culture robustness, it produces low lipid biomass typically laden with high levels of ash, which is problematic for conversion processes. When compared to more traditional biomass materials considered for conversion feedstocks, such as agricultural and woody residues, ATS biomass has unique challenges for conversion. ATS biomass typically contains high amounts of poorly defined ash (inorganic, non-convertible material) and organic compounds. However, ATS has been tested as a conversion feedstock for hydrothermal liquefaction (HTL) and fermentation [9,10] and found to be a promising feedstock. Of note, these conversion technologies are relatively insensitive to ash, when compared to pyrolysis or specific catalytic processes [11]. This makes them natural possibilities for the conversion of ATS material to bioenergy; however, ash is still a concern given that it must be transported, removed, and disposed of, and will likely have deleterious wear and abrasion effects on feedstock handling and conversion equipment [12,13].

Existing chemical preprocessing methods that target the removal of physiological ash components include low-severity water and dilute-acid leaching. Water washing at temperatures from ambient to near boiling can remove exogenous ash components such as sodium, alumina, iron and silica introduced via soil entrainment [14]. However, significant removal of physiological ash components, including the bulk of the alkaline earth and alkali metals that are inhibitory to pyrolysis conversions, via their catalysis of secondary cracking of vapors and reduction of bio-oil yield and quality, may require dilute-acid leaching [15–18]. Traditionally, these methods have been applied to agricultural and forest residues, which contain between one and 20 wt% ash. This total ash content is typically composed of 25% to 80% exogenous ash (e.g., entrained soils), with the remaining being physiological ash (e.g., intracellular nutrients or structural silica). In cases where it was desirable to remove high-levels of ash, such as from relatively dirty multi-pass corn stover, alkaline extractions have been applied with some success [19]. The use of alkali drastically increases the solubility of silica, which is typically the primary ash constituent in ATS systems due to its structural significance in diatoms and soils and clays embedded within filamentous algae [20–23].

The research presented here tested the efficacy of water washing and dilute-alkaline extractions as a potential preprocessing method to improve the feedstock quality attributes of biomass materials collected from ATS systems for potential conversion in HTL, biochemical, or other thermochemical conversion technologies.

It is important to note that any ash removal methods should ultimately be tailored, to the extent possible, to a given ATS system. Variables including the total ash, ash speciation, composition of physiological versus introduced ash, and composition of dissolved ash versus total suspended solids (TSS) could affect which approach or severity is most optimal. For this reason, both relatively mild and relatively severe conditions were tested in the present study. The conditions considered to be mild were carried out at atmospheric pressure and temperatures < 100 °C. The conditions considered to be severe were carried out in Parr reactors at temperatures > 100 °C, which required pressurization with nitrogen head to maintain liquid conditions in the reactor. Thus, two distinct types of systems with different requirements were tested. Equilibrium water washing and four concentrations of an alkali catalyst (NaOH) were tested at three different temperatures in low-severity batch conditions. In addition, equilibrium water washing and two concentrations of NaOH were tested at two different temperatures in high-severity batch conditions. The effects on soluble convertible material, ash, and ash composition are reported here and discussed with respect to their relevance to the convertibility of biomass material collected from ATS systems.

2. Material and methods

2.1. Sample collection

Algae was harvested from either a 0.61-meter-wide by 24.38-meter-long raceway (ATS 1) or a 0.30-meter-wide by 12.19-meter-long raceway (ATS 2). Both algal turf scrubbers received raw seawater directly from the Laguna Madre (Corpus Christi, TX) at a flow rate of ~189 L per minute with a dump bucket rate of 20 dumps per minute. The horizontal surfaces of both ATS systems were covered with 3-D matrices. The matrix on ATS 1 was a specially woven carpet InterfaceFLOR (La Grange, GA) described in Adey et al. [1], while the matrix on ATS 2 was Everlast Sequoia Fescue (Everlast Turf, Dallas, TX). Harvesting was accomplished by scraping the biomass from the substrate using a squeegee. A felt-lined mesh basket was placed at the end of each lane to collect the harvested biomass. Biomass was allowed to dewater for approximately 2 h before a sample was collected. Samples were placed into plastic ziplock bags and stored in Styrofoam coolers on ice for overnight shipment to Idaho National Laboratory. Homogenization was performed with a Ninja® blender (SharkNinja, Needham, MA) with 30 s pulses. Samples were then split into representative portions and stored at –20 °C until use.

2.2. Ash removal protocol at ambient conditions

An alkaline catalyst, NaOH, was used to improve the removal of ash constituents, especially soil- and structurally-embedded silica. Table 1 shows the test matrix used to determine the effects of catalyst concentration and temperature on the removal of organics during leaching.

Experiments were completed with triplicate replicates at each set of conditions. Results from each replicate were averaged, and the standard deviation across the three replicates was calculated. The figures in this manuscript show these average values (data points) and standard deviations (error bars). An initial set of experiments were carried out for 24 h to determine when the equilibrium extraction, as measured by the aqueous concentration of silicon and potassium in the extraction media, was achieved. Buffering capacity of the biomass was also determined by titration. One-mL aliquots of 50 wt% NaOH were added sequentially to a stirred batch reactor containing 5 wt% solids of the ATS biomass material. After each addition, the pH was allowed to equilibrate. Additionally, three randomly selected untreated samples were characterized to measure baseline total ash and ash composition for comparison against extracted material.

Extraction media were prepared by adding an alkaline catalyst (NaOH, Sigma Aldrich, USA) and deionized (DI) water to the required volumes, dependent upon the desired NaOH loading, in 250 mL Erlenmeyer flasks. A solids loading of 5 wt% was chosen based on previous biomass ash removal studies [12]. The Erlenmeyer flasks were then placed in temperature-controlled incubators (Innova 44, New Brunswick Scientific, USA). Calibrated thermometers were placed in the incubators to ensure temperature control accuracy. The samples were placed into flask holders attached to a platform that rotated at 120 rotations per minute.

Table 1

Experimental matrix used to construct a study on the effects of alkalinity and temperature on ash removal efficacy of biomass material collected from an algal turf scrubber.

| Sodium hydroxide loading (% db) | Tests at atmospheric pressure | Tests at 50 psig |
|---------------------------------|-------------------------------|------------------|
| Water wash | 25 °C, 50 °C, 80 °C | 130 °C |
| 0.5% NaOH | 25 °C, 50 °C, 80 °C | |
| 1.0% NaOH | 25 °C, 50 °C, 80 °C | 100 °C, 130 °C |
| 1.5% NaOH | 25 °C, 50 °C, 80 °C | |
| 2.0% NaOH | 80 °C | |

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