Data Article

Process evaluation data supporting studies on swing strategies to recover N-ethylbutylamine after wet lipid extraction from microalgae

Ying Du, Veronika Cyprichová, Kevin Hoppe, Boело Schuur*, Wim Brilman

University of Twente, Faculty of Science and Technology, Sustainable Process Technology Group, the Netherlands

ABSTRACT

In this paper, we publish information that has not been published before, but is needed to evaluate processes for wet lipid extraction from microalgae and recover the solvent N-ethylbutylamine (EBA), for example as presented in [1], the article entitled “Process evaluation of swing strategies to recover N-ethylbutylamine after wet lipid extraction from microalgae” in which we evaluate and interpret temperature swing and CO2-swing approaches. This includes selection of microalgae slurry concentration used in the extraction process, information on switching of EBA with CO2, data on the amount of EBA in solid residue after extraction, recoverability from the solid residue, and on recoverability of the solvent from the aqueous raffinate by liquid-liquid extraction and distillation of the solvent and EBA after the liquid-liquid extraction. Also information on phase behavior of binary mixtures of EBA and water is presented. Finally, detailed information on all flows in the process flow diagrams that are given in the article [1] is presented.

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1. Data

1.1. Comparison of lipid extraction yields and concentration costs for microalgae slurry concentrations of 5 wt.% and 10 wt.%

The algae concentration after centrifugal concentration by e.g. spiral plate centrifugal equipment (Evodos) is around 20 wt.% to 30 wt.%. For practical wet lipid extraction from microalgae with N-ethylbutylamine (EBA), the concentration should be reduced to e.g. 5 or 10 wt.% to reach a viscosity where liquid-liquid extraction is practical (see Fig. 1). In previous studies, 5 wt.% algae slurry was applied [2–4], here we present results using 10 wt.% which might be interesting to reduce size of equipment and possibly energy usage.

![Fig. 1. Microalgae concentration in different process steps.](image-url)
Extractions with switchable solvents (Scheme 1) were performed with non-stressed *Neochloris oleoabundans* algae in slurries of 5 wt.% and 10 wt.%, and for both 5 and 10 wt.% algae slurries, a similar lipid extraction yield was found (13.1 wt.% ± 0.2 wt.%, for the 5 wt.% slurry) and (13.0 wt.% ± 0.04 wt.% for the 10 wt.% slurry).

The difference in energy usage for concentrating algae slurry from 1 wt.% (algae concentration after harvesting) to 5 wt.% or 10 wt.% is calculated using Evodos dryer type 10 which has an energy requirement of 1.05 kWh/m³ [5], and was found to be 0.32 MJ/kg dry algae for 5 wt.% slurry and 0.36 MJ/kg dry algae for 10 wt.% slurry.

1.2. Quantification of the EBA content in solid residue by distillation

After lipid extraction, the solid paste was separated from the two liquid layers by centrifugation. From earlier work [4], it is known that algae cell walls stayed intact during lipid extraction, and that EBA is present in the cells after extraction, and most likely there is a significant amount of EBA lost in the paste around the cells. The loss of solvent to algae residues was determined by a distillation experiment in a short path distillation setup. The liquids were distilled from the algae cell residue after lipid extraction. The total liquid content of the algae paste that was obtained by filtration after extraction, was determined gravimetrically to be 92.0 ± 0.0 wt.%. The composition of the distillate was analyzed by GC-MS, showing that EBA counts for 15.2 ± 1.0 wt.% of the total mass of the wet residual paste while the remaining liquid was water.

1.3. Comparative study on wash solvents for washing EBA out of the solid residue

Four different solvents, i.e. cyclohexane, cyclohexane + water, water and ice water were investigated for EBA recovery from algae cells residue by washing. For cyclohexane, a 10:1 cyclohexane:paste ratio was applied, for cyclohexane/water in a 1:1 (w/w) mixture, as well as with water and ice-water a 20:1 ratio with the algae paste was applied. Results for EBA recovery efficiency of the four tested solvents are shown in Fig. 2.

Fig. 2 shows that the used solvents have single stage recovery performances ranging from around 70 to 90%. Using the combination of cyclohexane and water recovered more EBA than only using cyclohexane as extractant. Water also shows a higher recovery efficiency than the pure organic solvent cyclohexane. Using water as the extractant, a set of experiments was carried out with varying solvent/feed ratio and including multiple extraction steps. Four different solvent/feed ratios were used, which were 20:1, 15:1, 10:1 and 5:1. After mixing and liquid solid separation, fresh water was added into the algae paste residue for the next extraction step. Three extraction steps were performed. The results are shown in Fig. 3.

1.4. Extraction of EBA from the wash water followed by distillation of the extracting solvent and EBA

a) Single stage extraction and NRTL parameters

The results from the single stage extraction results of the EBA extraction from a saturated aqueous solution into an organic solvent are displayed in Fig. 4. To facilitate NRTL-parameter fitting for EBA and

![Scheme 1. Switching mechanism of secondary amines [7].](image)

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The phase behavior of water-EBA binary mixtures was measured for $298 < T/K < 347$, see Fig. 5. The NRTL parameters that were fitted to this dataset are listed in Table 1, while the binary interaction parameters for the other binary interaction pairs are listed in Table 2. The Sunflower oil – EBA interaction parameters were also not available and have been regressed to ternary EBA – water – sunflower oil data (see Fig. 6).

b) Multistage extraction

![Graph showing EBA recovery efficiency at varying solvent to feed ratio and three extraction steps.](image-url)
Table 1
NRTL binary parameters of EBA-water system.

| Component i | H$_2$O | Component j | EBA |
|-------------|--------|-------------|-----|
| $A_{ij}$    |        | $A_{ji}$    |     |
| $B_{ij}$    | 4.56   | $B_{ji}$    | 12.56|
| $\alpha$    | 111.14 |             |     |
| $\beta$     | 3960.38|             | 0.40|

Fig. 4. Single stage extraction efficiency for EBA recovery from water using different solvents.

Fig. 5. Phase diagram of EBA-water binary system. Symbols: experiments, line: NRTL model fit. The fitted parameters as listed in Table 1.
1.4.1. Process scheme

An overview of the refinery part where lipid depleted algae are washed, and the wash water is extracted in Fig. 7, an overview of streams 1–4 is given in Table 3. Based on a lipid extraction yield of 47 wt.% [3], 0.53 kg lipid depleted dry biomass is in #1 and leaves the system via #3. The Extract A from the water wash is extracted with a solvent, after which part of the water is discharged. For multistage extraction process design, the purity constraint for discharge of water is an important boundary condition. Furthermore, in case of bubbling with carbon dioxide, the properties of the solvent after switching is important, therefore this was experimentally validated (Fig. 10), and quantified (Tables 5 and 6).

1.4.2. Raffinate purity requirement

To be able to purge the wastewater, the EBA content should fulfill environmental legislation constraints after multistage extraction. To determine this constraint, the chemical oxygen demand (COD) was taken as measure. The following expression was used to calculate the COD concentration.

\[ C_6H_{15}N + 9O_2 \rightarrow 6CO_2 + 6H_2O + NH_3 \]
Table 3
Wash flows in washing EBA from raffinate and microalgae cells after wet lipid extraction normalized to the dry algae mass (stream numbers refer to streams in Fig. 7).

| Stream             | #1   | #2    | #3    | #4    |
|--------------------|------|-------|-------|-------|
| Lipid depleted algae (kg/kg dry algae) | 0.53 | –     | 0.53  | –     |
| Water (kg/kg dry algae)             | 5.09 | 132.50| 2.04  | 135.55|
| EBA (kg/kg dry algae)               | 1.01 | –     | –     | 1.01  |
| COD (mg/L)                       | –    | –     | –     | –     |

Table 4
Optimized (minimum reflux ratio) energy cost (MJ/kg contaminated water) for EBA recovery from the solvent by distillation using cyclohexane, dodecane and hexadecane in extraction columns.

| Solvent         | Cyclohexane | Dodecane | Hexadecane |
|-----------------|-------------|----------|------------|
| Solvent to feed ratio | 1:6         | 1:1.8    | 1:1.3      |
| Number of stages | 19          | 24       | 18         |
| Energy use without heat exchanger [MJ/kg contaminated water] | 0.0957 | 0.1353 | 0.1804 |
| Energy use with heat exchanger [MJ/kg contaminated water] | 0.0845 | 0.0396 | 0.0447 |

Table 5
Switching forward results of EBA in water.

| EBA: water (w/w) | 5:1 | 2:1 | 1:1 | 1:2 | 1:5 |
|------------------|-----|-----|-----|-----|-----|
| Time to form one phase (min) | 4   | 5   | 5   | 3   | 2   |

Table 6
Conversion percentage of EBA when reacts with CO2.

| Time (min) | 5 | 15 | 30 |
|------------|---|----|----|
| EBA conversion (%) | 25 | 48 | 69 |
Fig. 8. Simulation results of EBA content in raffinate at different solvent to feed ratios and extraction stages when using (A) cyclohexane, (B) dodecane and (C) hexadecane as extractant.
The COD of stream #4 was calculated to be $2.1 \times 10^4$, and for streams containing COD’s between 750mg/L and $2.5 \times 10^4$, legislation requires a 90% reduction in COD \cite{6}. This means that the COD concentration has to be lower than 2100 mg/L to meet the requirement for waste water disposal at the point of discharge. And that COD concentration corresponds to a EBA concentration of 740 mg/L. The multistage extraction should reduce the EBA concentration to below 740 mg/L.

1.4.3. Multistage extraction data

The multistage extraction process was simulated for three solvents, and the data is presented in Fig. 8 for the solvents cyclohexane, dodecane and hexadecane at various solvent to feed ratios. The horizontal line at 740 mg/L is the requirement for waste water at the point of discharge.

c) Distillation of extracting solvent and EBA

For each of the three solvents cyclohexane, dodecane and hexadecane, a distillation process was simulated to recover EBA from the minimum solvent flow as determined in Fig. 8, and the heat duty was minimized by searching the minimum reflux ratio RR for a number of simulations with varying number of stages. This procedure is plotted in Fig. 9 for the solvent with the minimum heat duty, dodecane. In Table 4, the minimum heat duty is expressed per kg of contaminated water (stream #8 in Fig. 7). The simulation of the entire process results in details on all streams given in Tables 7 and 8, for the two processes respectively. Properties of relevance for all solvents are given in Table 9.

The extraction column is designed under the condition that pure organic solvent is used as extractant. However, when a distillation is applied for regenerating dodecane and EBA from the extract, the distillate and bottoms product are not pure solvent anymore. This difference may influence the design of extraction column. In this study, the distillation target was set as a distillate that contains 99.9 wt.% EBA and a bottoms product that contains 99.9 wt.% dodecane. When this regenerated 99.9 wt.% dodecane is used as extractant, the extraction column has to be at least 39 stages in order to have the raffinate that can meet the point of discharge.

Fig. 9. Calculated results of NTS and RR for which the distillation purity target of 99.9 w% purity in both bottom (dodecane) and top (EBA) is reached.
Table 7
Flow table of steady-state operation of wet lipid extraction with EBA by temperature switching for processing 1 kg (dry weight) of Fresh water (FW) -stressed Neochloris Oleoabundans. This flow table corresponds with the flows indicated in Fig. 11 in Ref. [1].

| #1 | #2 | #3 | #4 | #5 | #6 | #7 | #8 | #9 | #10 |
|----|----|----|----|----|----|----|----|----|----|
| Lipid | 0.47 | 0 | 0 | 1.00 | 0 | 1.00 | 0.47 | 0.50 | 0.50 | 0 |
| Lipid depleted dry biomass | 0.53 | 0 | 0 | 0.53 | 0.53 | 0 | 0 | 0 | 0 | 0 |
| Water | 4.00 | 0 | 0 | 9.00 | 0 | 5.09 | 3.91 | 0 | 44.37 | 3.96 | 1.04 |
| EBA | 0 | 0 | 0 | 1.00 | 0 | 1.01 | 8.99 | 0 | 11.09 | 8.94 | 5.54 \( \times \) 10^{-2} |
| Dodecane | 0 | 0 | 0 | 0 | 1.01 \( \times \) 10^{-3} | 0 | 1.01 \( \times \) 10^{-3} | 0 | 0 | 0 | 0 |
| Total | 5.00 | 0 | 1.35 \( \times \) 10^{-3} | 20.53 | 6.63 | 13.91 | 0.47 | 55.96 | 13.40 | 1.10 |

| #11 | #12 | #13 | #14 | #15 | #16 | #17 | #18 | #19 | #20 |
|-----|-----|-----|-----|-----|-----|-----|-----|-----|-----|
| Lipid | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Lipid depleted dry biomass | 0 | 0.53 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| Water | 39.37 | 2.04 | 135.55 | 0 | 132.50 | 1.09 | 1.96 | 0 | 0 | 0 |
| EBA | 2.10 | 0 | 1.10 | 0 | 9.17 \( \times \) 10^{-2} | 7.53 \( \times \) 10^{-4} | 1.35 \( \times \) 10^{-3} | 1.08 | 7.60 \( \times \) 10^{-2} | 1.00 |
| Dodecane | 0 | 0 | 0 | 1.01 \( \times \) 10^{-3} | 0 | 0 | 0 | 75.96 | 75.95 | 1.01 \( \times \) 10^{-3} |
| Total | 41.47 | 2.57 | 136.64 | 1.01 \( \times \) 10^{-3} | 132.59 | 1.09 | 1.96 | 77.04 | 76.03 | 1.01 |

1.5. CO₂ induced EBA switching in presence of water

In this section we present relevant detailed data for the switching of EBA with CO₂.

1.6. Process flow tables

2. Experimental design, materials and methods

2.1. Comparison of lipid extraction yields and concentration costs for microalgae slurry concentrations of 5 wt.% and 10 wt.%

2.1.1. Chemicals

FW-stressed Neochloris oleoabundans algae (under nitrogen limitation condition) were obtained from AlgaePARC (NL). Algae paste was mixed with water to get 5 wt.% or 10 wt.% algae slurry. The water content in algae slurries was determined by weighing a sample before and after drying at 105 °C for 24 h.

2.1.2. Experiments

Extraction experiments with 5 wt.% slurries and 10 wt.% slurries were carried out by mixing with EBA for 18 h at 298 K and a solvent to feed ratio of 1:1 (mass). After phase separation by centrifuge, the EBA-lipid phase was isolated and CO₂ was bubbled in a flow rate of 2 VVM for 60 min, during which the solvent switched in to the hydrophilic form and separated from hydrophobic lipid. After collecting the lipid and drying, the lipid yield was determined by weighing. The method was previously described in detail [2,3].

2.2. Quantification of the EBA content in solid residue by distillation

2.2.1. Chemicals

Neochloris oleoabundans algae paste obtained after extraction with EBA and centrifugation.

2.2.2. Experiments

The EBA content of algae paste after lipid extraction was measured by a drying/distillation experiment and followed by GC-MS measurement. A known amount of algal cells residue after lipid extraction and filtration were transferred into a 50 mL flask for distillation (see Fig. 13). The flask was heated up to 125 °C in an oil bath at atmospheric pressure. The vapors were collected in a condenser which was cooled in ice. The amount of liquid collected was measured gravimetrically. The liquid
Table 8
Flow table of steady-state operation of wet lipid extraction with EBA by CO₂ switching for processing 1 kg (dry weight) of FW-stressed Neochloris Oleoabundans. This flow table corresponds with the flows indicated in Fig. 12 in Ref. [1].

| Unit [kg]           | #1   | #2   | #3   | #4   | #5   | #6   | #7   | #8   | #9   | #10  | #11  | #12  |
|---------------------|------|------|------|------|------|------|------|------|------|------|------|------|
| Lipid               | 0.47 | 0    | 0    | 0.47 | 0    | 0.47 | 0    | 0.47 | 0    | 0    | 0    | 0    |
| Lipid depleted      | 0.53 | 0    | 0    | 0.53 | 0    | 0.53 | 0    | 0    | 0    | 0    | 0    | 0    |
| dry biomass         |      |      |      |      |      |      |      |      |      |      |      |      |
| Water               | 4.00 | 0    | 0    | 9.00 | 5.09 | 3.91 | 0    | 0    | 9.13 | 0    | 0    | 0    |
| EBA                 | 0    | 0    | 0.54 | 10.00| 1.01 | 8.99 | 0    | 0    | 6.91 | 0    | 0    | 0    |
| EBA carbamate       | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 2.19 | 0    | 0    | 0    |
| EBA bicarbonate     | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0.82 | 0    | 0    | 0    |
| CO₂                 | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    |
| N₂                  | 0    | 0    | 0    | 0    | 0.61 | 0    | 0    | 0    | 0    | 9.99 | 9.99 | 0    |
| Dodecane            | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    |
| Total               | 5.00 | 0.54 | 0    | 20.00| 6.63 | 13.38| 0.47 | 0.47 | 19.05| 9.99 | 9.99 | 0.49 |

| Unit [kg]           | #13  | #14  | #15  | #16  | #17  | #18  | #19  | #20  | #21  |
|---------------------|------|------|------|------|------|------|------|------|------|
| Lipid               | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    |
| Lipid depleted      | 0    | 0    | 0    | 0    | 0    | 0.53 | 0    | 0    | 0    |
| dry biomass         |      |      |      |      |      |      |      |      |      |
| Water               | 3.71 | 3.71 | 0    | 1.29 | 4.22 | 2.04 | 135.55| 0    | 132.50|
| EBA                 | 8.39 | 8.39 | 0    | 6.85×10⁻²| 0.22 | 0    | 1.10 | 0    | 9.17×10⁻²|
| EBA carbamate       | 0.66 | 0    | 0.66 | 0    | 0    | 0    | 0    | 0    | 0    |
| EBA bicarbonate     | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    |
| CO₂                 | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    |
| N₂                  | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    |
| Dodecane            | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    | 0    |
| Total               | 12.76| 12.10| 0.66 | 1.35 | 4.44 | 2.57 | 136.64| 1.01×10⁻³| 132.59|

| Unit [kg]           | #22  | #23  | #24  | #25  | #26  |
|---------------------|------|------|------|------|------|
| Lipid               | 0    | 0    | 0    | 0    | 0    |
| Lipid depleted      | 0    | 0    | 0    | 0    | 0    |
| dry biomass         |      |      |      |      |      |
| Water               | 1.09 | 1.96 | 0    | 0    | 0    |
| EBA                 | 7.53×10⁻⁴| 1.35×10⁻³| 1.08 | 7.60×10⁻²| 1.00 |
| EBA carbamate       | 0    | 0    | 0    | 0    | 0    |
| EBA bicarbonate     | 0    | 0    | 0    | 0    | 0    |
| CO₂                 | 0    | 0    | 0    | 0    | 0    |
| N₂                  | 0    | 0    | 0    | 0    | 0    |
| Dodecane            | 0    | 0    | 75.96| 75.95| 0    |
| Total               | 1.09 | 1.96 | 77.04| 76.03| 1.01 |
| Solvent       | Cyclohexane | Hexane | Heptane | Dodecane | Hexadecane | DCM |
|--------------|-------------|--------|---------|----------|------------|-----|
| Boiling temperature (°C) | 80.55 | 67.85 | 97.85 | 216.20 | 286.90 | 39.85 |
| Solubility in water at 25°C (mg/L) | 55.00 | 9.50 | 2.93 | 3.70 \times 10^{-3} | 2.10 \times 10^{-5} | 13200 |
| ΔH_{vap} (kJ/kg) | 393.30 | 359.71 | 359.25 | 361.16 | 359.24 | 330.00 |
| Specific heat capacity (kJ/(kg·°C)) | 1.85 | 3.08 | 2.24 | 2.21 | 2.26 | 1.21 |

**Fig. 10.** Gel formation and solid liquid separation of EBA/water mixture after 2 h CO\(_2\) bubbling.

**Fig. 11.** \(^{13}\)C NMR spectra of reacted EBA (both solid and liquid phase).
sample was diluted in acetone in the ratio of 1:20 and then measured by GC-MS for its EBA content. Each experiment was performed at least four times.

GC–MS analyses were performed using a 7890A Agilent HP gas chromatograph equipped with an Varian CP 9154 capillary column (60 m, 250 μm i.d., 0.25 μm film thickness), connected to a 5975C Agilent HP quadrupole mass spectrometer. The injection temperature was 250 °C and the detector temperature was 280 °C. Helium was used as carrier gas at a constant pressure of 2.28 bar. Mass spectra were recorded under electron ionization (70 eV) at a frequency of 2.5 scan per second within the range 12–600 m/z. The oven was programmed as follows: 45 °C for 4 min, ramps of 5 °C/min to 280 °C where the temperature was held for 10 min. The total analysis time was about 61 min.

Standard solutions from 1 to 80 wt.% of EBA in demineralized water were prepared and diluted in acetone in the ratio of 1:20 for calibration of the GC–MS.

2.3. Comparative study on wash solvents for washing EBA out of the solid residue

2.3.1. Chemicals

*Neochloris oleoabundans* algae paste obtained after extraction with EBA and centrifugation, milli Q water, cyclohexane (99.5%, Sigma-Aldrich), HCl (Reag. Ph. Eur., Fluka),..
2.3.2. Experiments

EBA recovery from algae paste through washing was studied with four different solvents, i.e. cyclohexane, cyclohexane + water, water and ice water. In all cases, washing was performed for at least 18 h. The solvent/feed ratio was 10:1 when cyclohexane was used and the ratio was changed to 20:1 when cyclohexane + water (1:1 w/w), water and ice water were used. EBA concentrations in water were measured by titration with HCl, in cyclohexane by GC-MS as described in Section 2. In the titration, a Metrohm 785 DMP Titrino probe was used, connected with Metrohm 806 Exchange unit. HCl (0.1 M) was used as titrant.

2.4. Extraction of EBA from the wash water

2.4.1. Chemicals

EBA (>98.0%, Aldrich), acetone (>99.5%, Sigma-Aldrich), cyclohexane (99.5%, Sigma-Aldrich), dodecane (>99%, Sigma-Aldrich), dichloromethane (DCM) (>99.8%, Sigma-Aldrich), hexadecane (99%, Sigma-Aldrich), hexane (>95%, Sigma-Aldrich), HCl (Reag. Ph. Eur., Fluka), milli Q water.

2.4.2. Experiments

In the EBA extraction experiments, cyclohexane, hexane, heptane, dodecane, hexadecane and DCM were equilibrated with an aqueous phase saturated with EBA. Different solvent to feed ratio (1:10, 1:5, 1:2 and 1:1) were applied. The EBA amount in aqueous phase before and after mixing was measured by pH titration.

2.4.3. Parameter fitting and simulations

Simulations of multistage countercurrent extractions in columns were done using Aspen Plus V8.8, and nonrandom, two-liquid (NRTL) model was used in the simulations. The required binary interaction parameters were taken from the Aspen Plus database for the binary pairs cyclohexane — water, dodecane — water, and hexadecane — water. For EBA — water the parameters were not available, and the binary interaction parameters were fitted to experimental phase equilibrium of the EBA — water binary mixture.

Using RadFrac in Aspen Plus, the distillation column to recover the EBA from the extraction solvent was simulated at atmospheric pressure. For each of the solvents cyclohexane, dodecane and hexadecane, the number of stages and the reflux ratio were varied to find the minimum heat duty for the reboiler. Results were tabulated both for distillation after extraction without heat exchanger, and for a process configuration where a heat exchanger was applied to the heat the feed stream with a temperature difference of 10 °C before entering the distillation column.

2.5. CO2 induced EBA switching in presence of water

2.5.1. Chemicals

EBA (>98.0%, Aldrich), milli Q water, HCl (Reag. Ph. Eur., Fluka).

2.5.2. Experimental methods

Pure CO2 in a flow rate of 5 VVM was bubbled through the mixture of EBA and water (in different mass ratio of 5:1, 2:1, 1:1, 1:2 and 1:5) at ambient conditions.

The conversion percentage of EBA in an EBA-water (1:1 w/w) mixture was measured by titration with HCl. In the titration, a Metrohm 785 DMP Titrino probe was used, connected with Metrohm 806 Exchange unit. HCl (0.1 M) was used as titrant.

CO2 was bubbled to an EBA-water (1:1 w/w) mixture for 2 h to get complete gel formation and then separated into a solid and liquid phase by vacuum filtration and identified by 13C NMR. Quantitative 13C-NMR measurements were performed with a Bruker 400 MHz Ascend NMR spectrometer. Samples were prepared by dissolving approximately 0.05—0.1 g of sample in 1.0 mL of acetonitrile. Measurements were performed using “inverse gate decoupling” method with 5 seconds of relaxation time and 1024 scans. The acetonitrile signal was used as internal reference.
Manual integration of the spectra was performed with MestReNova software (version 6.1.1-6384). When present, solvent peak of acetonitrile was manually integrated and subtracted from the corresponding region. Based on the research of Böttinger et al. [8] and Vanderveen et al. [9], the peak associated with the bicarbonate ion appears near 160–162 ppm, while the peak associated with the carbamate ion appears near 163–165 ppm.

2.6. Process simulation

The various process steps were simulated in Aspen Plus, and the total heat duty was calculated and expressed per kg lipid.

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Conflict of interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.104416.

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