Research Article

Recovery and liquefaction of nitrogen-containing component and minerals from food processing wastes of vinegar using subcritical water

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Abstract
In recent years, environmental problems such as production of industrial waste are getting worse in Japan. In particular, food processing residues are typically disposed of by spending a large amount of money. This paper studied the treatment of two by-products from vinegar production (rice bran and sake lees) using subcritical water and analyzing the extracted solution for functional ingredients. The results from sake lees showed that an operating temperature of 180 °C and a reaction time of 30 min solubilized 85% of the nitrogen-containing component (mainly protein) from the raw material into water-soluble peptides. When rice bran was used as the raw material the solubilization rate of the nitrogen-containing components was greatly decreased at a reaction temperature of 190 °C or higher. It was shown that calcium in the raw material caused the formation of water-insoluble complexes with amino acids and ammonia such as $CaCN_2$. Subcritical water treatment was shown to be a useful technique to recover useful water-soluble components from residual solid biomass.

Keywords Biomass · Green chemistry · Subcritical water extraction · Hydrolysis

1 Introduction

Pollution due to the environmental discharge of residual organics from food processing has become a social problem all over the world. In recent years, the concept of technical innovation which is converting conventional chemical processes to more sustainable and 'green' procedures [1], has been pursued in Japan. The depletion of energy resources can be minimized by efficient utilization and conversion of waste to energy. The 'Basic Law for Promotion of Utilization of Biomass' was introduced in Japan in 2009. It is based on the philosophy of diversifying energy supply sources, and consideration for environmental conservation, and in more specific terms, it promotes biomass utilization.

Figure 1 shows the relationship between biomass conversion technology and products providing a template for value added [2]. Biomass resources are roughly classified into resource crops and waste processes in Japan. Some examples of food recycling technology are the production of fertilizer, oils and fats from food waste.

Examples of food recycling technology include the production of fertilizer from food waste for use by farmers, and the application to fats and oils and fat products.

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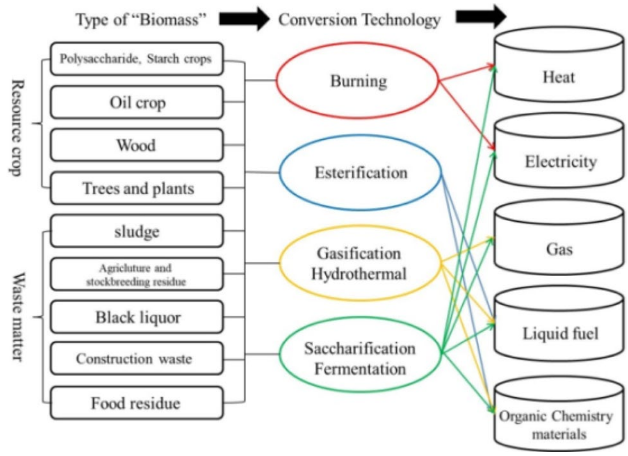

Fig. 1 Biomass conversion technologies and their products

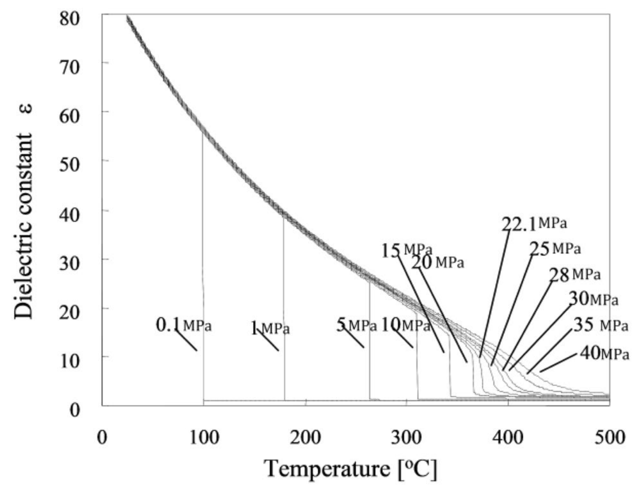

Fig. 2 The ion product of water

For instance, in 2017, Japan produced 584,000 tons of rice bran and sake lees as food processing residues compared to the 78,000 tons produced in 2016 (Maruboshi vinegar production was 1 ton of rice bran, and 15 tons of sake lees per year in 2017) [3–6]. Efficient recycling technology would not only reduce the volume of solid waste generated but also reduce the costs paid by companies for waste disposal, thus increasing revenue. Hence, wide spreading of the use of this concept will have a positive effect in the economy of the country. Furthermore, vinegar industries can obtain high value-added vinegar by adding acetic acid to the subcritical water extract and brewing it again. This process is regarded as an epoch-making technology since the residues of vinegar production is not discarded. Furthermore, in the vinegar industry, by adding acetic acid to the subcritical water extract and brewing it again, there is a possibility that it can be reused as high value-added vinegar. This is an epoch-making technology because the vinegar residue is not discarded.

The term subcritical water, refers to a state of water before reaching the critical state, meaning that pressure and temperature conditions are lower than its stablished critical point. At this point, the state of matter is determined by pressure and temperature conditions. Substances have intrinsic intermolecular distances, and phase changes occur under substance-specific conditions. However, there is a point at which it does not liquefy even if it is compressed above a certain temperature; this is called the critical point.

The critical temperature of water is 374.2 °C, and its critical pressure is 22.1 MPa, water at temperature and pressure conditions beyond these conditions is called supercritical water. This state combines the diffusibility of a gas and the reactive properties of a liquid and can be described as high-density gas. In subcritical water, molecules of water with high thermal energy due to high temperature are in a liquid state due to high pressure; it has both the properties of the gas that can enter any (narrow intermolecular gap) and like liquids, possesses the property to melts things. Figure 2 shows the ion product of water.

The ionic product constant (KW) of subcritical water is about 1000 times higher than in its normal state and ion dissociation of water occurs easily. In addition, the relative dielectric constant also has a high value of about 20–50, which increases its solvent capacity to the point that it is as soluble as alcohol and is easily hydrolyzed. Water molecules in this state have been reported to exhibit a catalytic function only by bias of charge [7]. Water in this state has the ability to act as an acid or an alkaline catalyst and is able to accelerate the hydrolysis reaction. Even in a high temperature region exceeding its critical temperature, the ion product can take a high value under a sufficiently high-pressure condition of 40–100 MPa.

Subcritical water can catalyze various chemical reactions; for instance, it hydrolyzes the cellulose in rice straw and chaff to form low-molecular-weight, solubilized compounds used for bioethanol fermentation [8–13], and can also solubilize sewage sludge [14–21]. By utilizing this feature, it is possible to create techniques for producing useful components in an environmentally friendly manner without using conventional chemicals. (i.e. organic solvents, inorganic acids, and expensive metal catalysts.)

Application of subcritical water to the solubilization and hydrolysis of proteins and sugars in food processing residues is a greener reaction technology that has the potential to enhance the effective utilization of biomass and reduce solid waste. This study focuses on rice bran and sake; which are high-volume food processing residues and investigates its treatment with subcritical water and nitrogen-containing compounds to extraction the mineral components (K, Ca, Mg) at high concentrations.

2 Experimental section

2.1 Materials

Rice bran and sake lees provided by Maruboshi vinegar corporation (Fukuoka) were used as raw materials, as shown in Fig. 3.

Their chemical compositions are shown in Table 1. Distilled water was used as a solvent in the subcritical water treatment. Standard substances used in the quantitative analysis of amino acids are also shown in Table 1.

2.2 Experimental apparatus and procedure

An autoclave shown in Fig. 4 was used to treat biomass residues in water at high temperatures and high pressures.

The reactor was constructed from SS 316 steel and has an internal volume of 500 mL. The reactor was charged with 45 g of raw material and 300 mL of distilled water, mixed with a stirrer, and then sealed. Thereafter, the temperature was raised to a predetermined setpoint (160–225 °C) by a band heater installed in the reactor. The heating time was 15 to 30 min. After reaching the predetermined temperature, the contents were reacted for 15–120 min while stirring at 300 rpm. The pressure in the reactor varied from 1.3 to 2.6 MPa depending on the vapor pressure of water and the product gas evolved during processing. After the subcritical water treatment, the band heater was removed from the reactor and a fan was used to quickly quench the reactor. After the reaction solution was sufficiently cooled (hereinafter this solution will be referred to as a sub-critical water treatment solution) was collected and separated into filtrate and water-insoluble components by suction filtration.

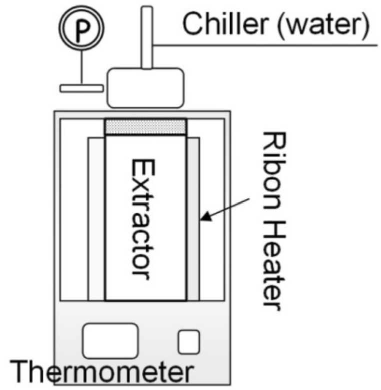

Fig. 4 Schematic diagram of experimental equipment for sub-critical water treatment

2.3 Analysis

The concentrations of amino acids, nitrogen, phosphorus, and minerals contained in the subcritical water treatment solution were quantitatively analyzed.

2.3.1 Amino acids

Amino acids were derivatized with OPA(o-Phthalaldehyde by Wako) and FMOC(9-Fluorenylmethyl Chloroformate by Wako) and separated by a column (2.6μ EVO C18 100×3 mm by Kinetex) for ultrahigh-speed analysis and then analyzed by High performance liquid chromatography (HPLC) with a fluorescence detector (NEXERA X 2 manufactured by SHIMADZU). Sixteen amino acids were isolated, as shown in Table 2.

Fig. 3 Raw materials used in this study: (**a**) Rice bran, and (**b**) Sake lees

Table 1 Composition of sake lees and rice bran

	Moisture content [%]	TN [ppm-N]	Water soluble amino acid amount [ppm-N]	TOC [ppm-C]	Ammonic nitrogen content [ppm-N]	Mineral amount [ppm]
Rice bran	9.69	1900	43.6	8.485	4.2	23.9
Sake lees	56.2	2510	127.1	8.835	21.3	96.5

Table 2 Amino acids quantitatively analyzed in this study

1. Aspartic acid	2. Glutamic acid	3. Serine	4. Histidine
5. Glycine	6. Threonine	7. Arginine	8. Alanine
9. Tyrosine	10. Valine	11. Methionine	12. Phenylalanine
13. Isoleucine	14. Leucine	15. Lysine	16. Proline

2.3.2 Total nitrogen concentration

Quantitative determination was made by dividing nitrogen into total nitrogen and ammonia-nitrogen, and the Kjeldahl method [22] was used for total nitrogen.

2.3.3 Ammonia nitrogen concentration

Analysis of ammonia-nitrogen was carried out using the Indophenol method [23].

2.3.4 Phosphorus content

Phosphorus content was determined using the molybdenum blue method [24].

2.3.5 Minerals

Three minerals: potassium, calcium and magnesium were quantitatively determined by atomic absorption spectrometry (AA-7000 by SHIMADZU).

2.4 Definitions of formulas

2.4.1 Solubilization ratio of raw material

$$\text{Raw material solubilization ratio} = \text{water} - \text{soluble nitrogen yield} = \text{TN concentration [ppm} - \text{N]}/\text{total amino acid concentration treated with HCl [ppm} - \text{N]} \quad (1)$$

2.4.2 Water-soluble peptide yield

$$\text{Water\_Soluble Peptide Yield} = \text{Water\_Soluble Nitrogen Yield} - \text{Amino Acid Yield Ammonia Yield} \quad (2)$$

2.4.3 Amino acid yield

$$\text{Amino acid yield} = \text{Amino acid concentration [ppm} - \text{N]}/\text{HCl Total amino acid concentration treated [ppm} - \text{N]} \quad (3)$$

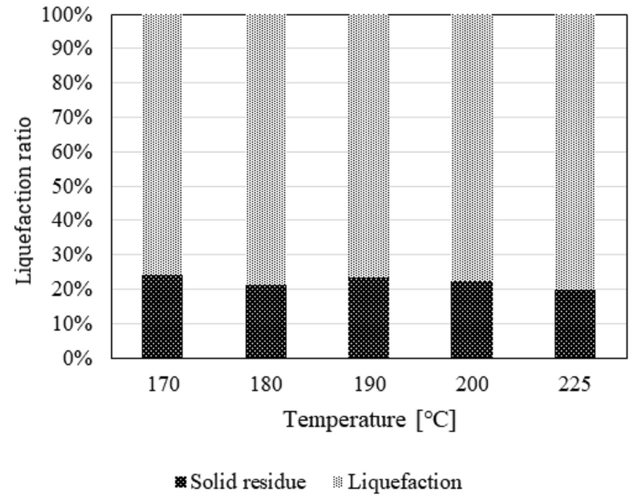

Fig. 5 Effect of temperature on the liquefaction strength-ratio of sake lees through the subcritical water treatment for 30 min

2.4.4 Ammonia yield

$$\text{Ammonia yield} = \text{ammonia} - \text{nitrogen concentration [ppm} - \text{N]}/\text{HCl Total amino acid concentration treated [ppm} - \text{N]} \quad (4)$$

2.4.5 Total organic carbon (TOC)

$$\text{TOC} = \text{Total Carbon (TC)} - \text{Inorganic Carbon (IC)} \quad (5)$$

2.4.6 Mineral amount

$$\text{Mineral amount} = \text{K concentration} + \text{Ca concentration} + \text{Mg concentration} \quad (6)$$

3 Results and discussion

3.1 Subcritical water treatment of sake lees

3.1.1 Effect of temperature on the liquefaction strength ratio

Relationship between the liquefaction rate of sake lees and operating temperature on the subcritical water treatment for 30 min was investigated, and the results are shown in Fig. 5. The liquefaction rate was in the range of 75–80% in spite of a temperature change from 170 to 225 °C.

3.1.2 Effect of temperature on the recovery of organic and inorganic compounds

We decided to consider the total concentrations of peptides as total amino acids. Figure 6a shows the results of comparing the water-soluble peptides, total amino acids, and ammonia-nitrogen when the treatment time was kept constant for 30 min and the sake lees were treated at a treatment temperature of 170–225 °C. The data shown in Fig. 6a suggested that the water-soluble peptides were degraded and decreased with increasing temperature, and that amino acids could be recovered more efficiently at low temperature.

It was noted that the amino acids increased again at 225 °C. Amino acids are produced by decomposition of proteins and peptides which are then decomposed into ammonia, and the yield is determined from these two factors. An increase in the amount of amino acids measured at 225 °C is conceivably caused by the decrease of the water-soluble peptide concentration exceeded by the ammonia concentration. The concentration of total amino acids released from sake lees were higher than that of rice bran, the details will be later discussed. A possible reason for this is that sake lees are obtained downstream of the rice bran in the vinegar process, so any proteins therein were more likely to be decomposed into amino acids.

Figure 6b shows the temperature and time dependence of the released phosphoric acid, phosphorus, and minerals from sake lees on subcritical water treatment. Use of subcritical water treatment under appropriate operating conditions made it possible to extract these minerals from sake lees into the aqueous phase. In addition, it was found that phosphorus present in sake lees can be recovered by appropriate temperature adjustment.

3.1.3 Effect of treatment time on the hydrolysis of protein fraction and minerals

Figure 7a shows the nitrogen balance in water-soluble organic compounds after treatment with subcritical water using sake lees as a raw material. Approximately 90% of protein was successfully converted into water-soluble peptide, amino acids, and ammonia after 30 min at >160 °C. This additional study investigated suitable operating conditions under which water-soluble nitrogen-containing components can be obtained from sake lees. Nitrogen-containing components were classified as protein, free amino acids, uric acid, nitrate nitrogen, and ammonia-nitrogen. During subcritical water treatment, the peptide bond of the water-insoluble peptide in the raw material is converted into a water-soluble peptide or amino acid.

Figure 7b shows the time dependence of phosphoric acid, phosphorus, and minerals of sake lees in subcritical water treatment at 180 °C. By conducting the subcritical water treatment under appropriate operating conditions, it was possible to extraction minerals from the sake lees into the aqueous phase without measurable loss.

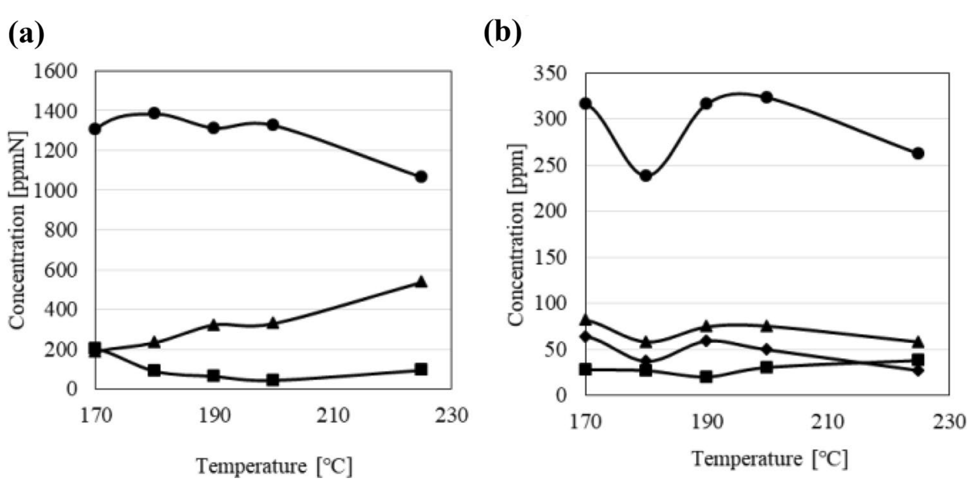

Fig. 6 Behavior of the nitrogen-containing components obtained after the subcritical water treatment of sake lees at 180 °C for 30 min: (**a**) water-soluble peptides, amino acids and ammonia (Symbols: (black circle) water-soluble peptides, (black square) total amino acids, and (black up-pointing triangle) ammonia); (**b**) phosphorus (all phosphorus derived from phosphoric acid) and minerals (Symbols: (black circle) phosphorus, (black up-pointing triangle) potassium, (black square) calcium, and (black diamond suit) magnesium)

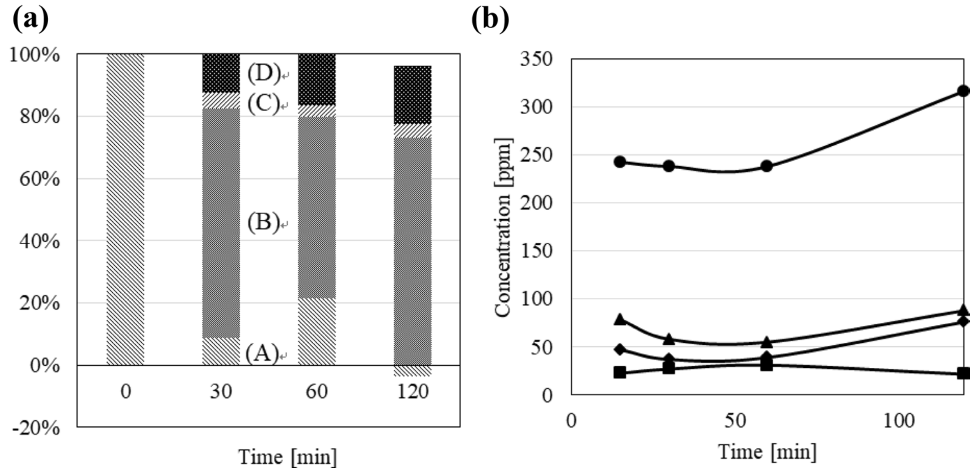

Fig. 7 Time course of the nitrogen-containing components obtained after the subcritical water treatment of sake lees at 180 °C: (**A**) organic compounds and ammonia ((a) water-insoluble protein, (b) water-soluble peptides, (c) total amino acids, and (d) ammonia); (**B**) phosphorus and minerals (Symbols: (black circle) phosphorus, (black up-pointing triangle) potassium, (black square) calcium, and (black diamond suit) magnesium)

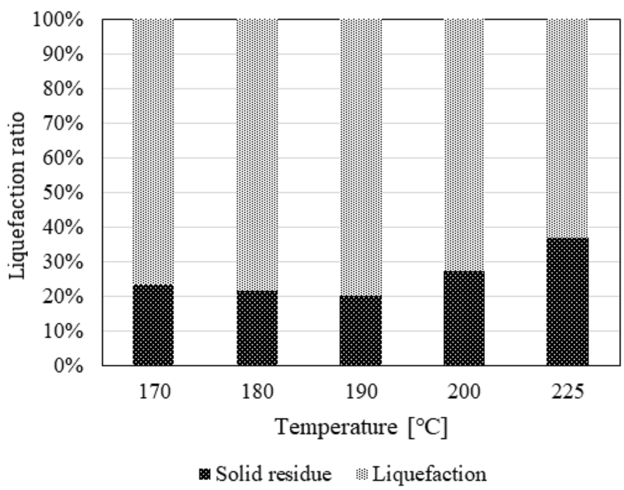

Fig. 8 Effect of temperature on the liquefaction strength ratio of rice bran through subcritical water treatment for 30 min

3.2 Subcritical water treatment of rice bran

3.2.1 Effect of temperature on the liquefaction strength ratio

The relationship between the liquefaction rate of rice bran and operating temperature on the subcritical water treatment for 30 min was investigated. Figure 8 shows that the liquefaction rate in the range of 170–190 °C was almost the same level (about 80%). The rate was reduced with increasing operating temperature most probably due to the combination of hydrolysis products that were formed during the subcritical water treatment.

3.2.2 Effect of temperature on the recovery of organic and inorganic compounds

Figure 9a compares the water-soluble peptides, total amino acids and ammonia-nitrogen released from rice bran after 30 min at a treatment temperature of 170–225 °C. Water-soluble peptides were degraded and

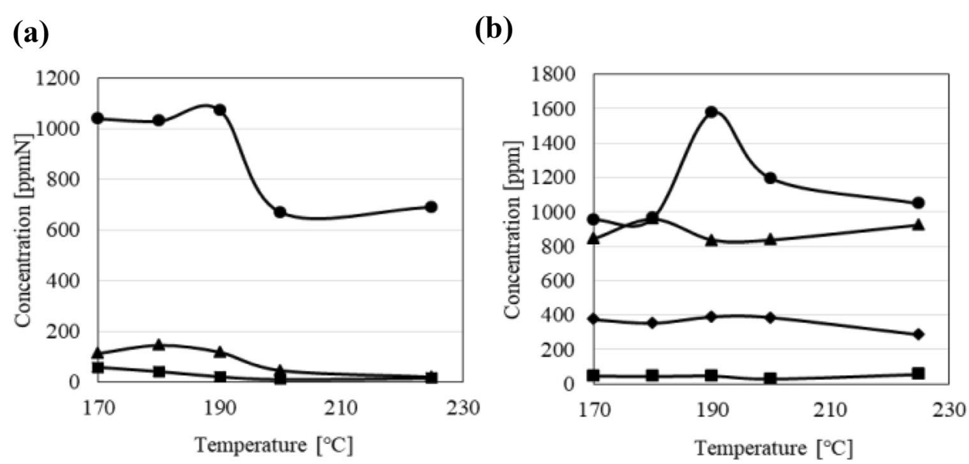

Fig. 9 Temperature dependence on the elution profile of functional ingredients after 30 min of exposure to subcritical water: (**a**) water-soluble peptides, amino acids and ammonia (Symbols: (black circle) water-soluble peptides, (black square) total amino acids, and (black up-pointing triangle) ammonia); (**b**) phosphorus and minerals (Symbols: (black circle) phosphorus, (black up-pointing triangle) potassium, (black square) calcium, and (black diamond suit) magnesium)

decreased in concentration with increasing temperature which suggests that the amino acids could be more effectively recovered at lower temperatures.

Ammonia increased due to decomposition of water-soluble peptides and amino acids in the case of sake lees as shown in Fig. 6a, but in the case of rice bran, which is rich in minerals, the minerals and ammonia reacted to become solids, such as Mg_3N_2 and $CaCN_2$. In fact, when treated at 225 °C, many solid residues remained. Total amino acid concentration was found to be lower in rice bran compared to sake lees. We consider the reason for this to be that the rice bran is generated upstream from sake lees in the manufacturing process of vinegar, so the protein contained in this residue was not as easily decomposed into amino acids.

Figure 9b shows the temperature and time dependence of the release of phosphoric acid, phosphorus, and minerals from rice bran by subcritical water treatment. Comparison between Figs. 6b and 9b indicate that phosphorous and minerals were more abundant in rice bran than in sake lees. It is considered that these large amounts of phosphorus and minerals may have reacted with ammonia to form a solid as discussed in Sect. 3.2.1.

3.2.3 Effect of treatment time on the hydrolysis of protein and mineral fractions

Rice bran was treated with subcritical water to obtain a liquefaction rate of about 88% at 30 min and 60 min (see Fig. 10a). Treatment for 120 min resulted in carbonization and high recovery of solid residue. It is suggested that the low liquefaction of rice bran is due to its lower water content, which promoted carbonization instead. In addition, water-soluble peptides, total amino acids, and ammonia decreased with treatment time. Water-soluble peptides and amino acids were suggested to be over-degraded due to an increase in subcritical water treatment time. The ammonia in the rice bran behaved differently than that in sake lees. It is suggested that higher mineral content in the rice bran reacted with ammonia to form solids such as magnesium nitride Mg_3N_2 and lime nitrogen $CaCN_2$. The concentrations of potassium and calcium did not change with increasing treatment time while the concentration of phosphorus gradually increased with time up to 1 h and that of magnesium was decreased within 0.5 h (see Fig. 10b).

3.3 Effect of feedstock ratio on component composition

As shown previously, the liquefied aqueous solution from sake lees contained a high concentration of amino acids and the one from rice bran had a large amount of minerals. In order to produce a high-quality liquefaction product, we investigated subcritical water treatment (180 °C, 30 min) using three mixing ratios of rice bran (R) to sake lees (S) in the feedstock (R/S = 3/1, 1/1 and 1/3). The total amino acid concentration in the mixed aqueous solution increased proportionally with the R/S ratio, as shown in Fig. 11a and b, and the concentrations of minerals showed a similar trend. These results indicate that there is no interaction between organic and inorganic compounds produced despite the starting materials being mixed and treated in subcritical water. This finding clearly indicates that a desired composition of amino acids and minerals can be prepared by the appropriate mixing of these two agricultural feedstocks.

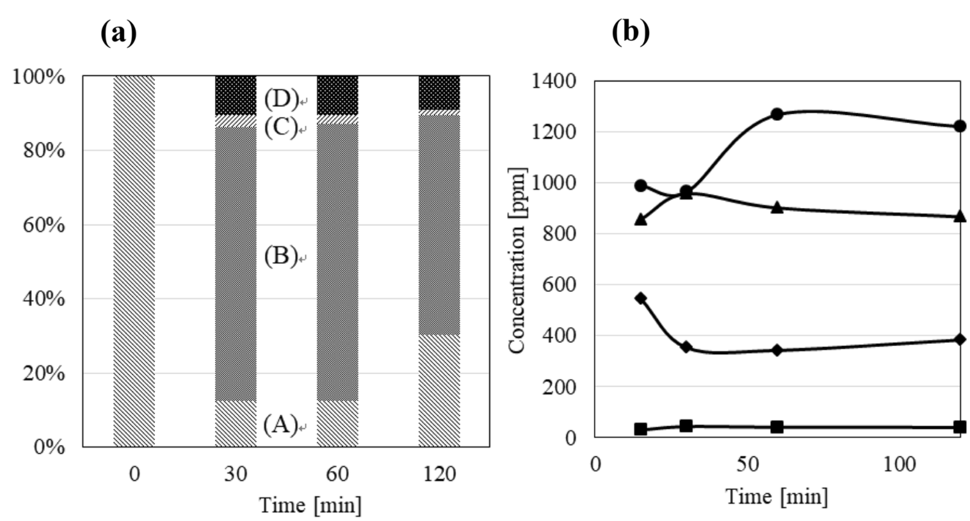

Fig. 10 Time course of the elution of nitrogen-containing components obtained from the subcritical water treatment of rice bran at 180 °C: (**A**) organic compounds and ammonia ((a) water-insoluble protein, (b) water-soluble peptides, (c) total amino acids, and (d) ammonia); (**B**) phosphorus and minerals (Symbols: (black circle) phosphorus, (black up-pointing triangle) potassium, (black square) calcium, and (black diamond suit) magnesium)

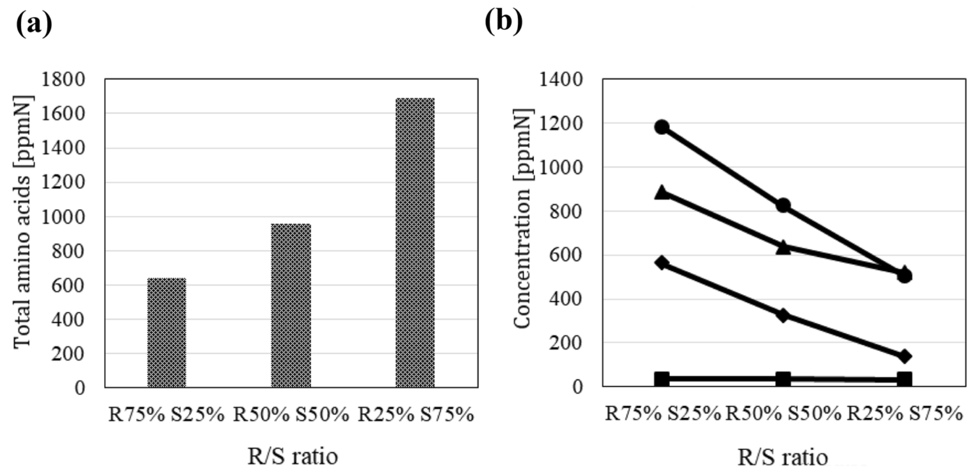

Fig. 11 Effect of the material ratio of Rice bran to sake lees (R/S) on the concentration of total amino acids (**a**) and the concentration of phosphorus and minerals (**b**). (Symbols: (black circle) phosphorus, (black up-pointing triangle) potassium, (black square) calcium, and (black diamond suit) magnesium)

4 Conclusions

In this study, subcritical water treatment was applied to two biomass-based food processing residues: rice bran and sake lees. Analysis was conducted on the inclusion and recovery of functional ingredients. It was shown that phosphorus and minerals can be recovered from rice bran, and amino acids and nitrogen can be recovered from sake lees. Simultaneous recovery of valuable components can be achieved by subjecting a mixed feedstock to subcritical water treatment using distilled water as a solvent.

Future studies will investigate the addition of acetic acid bacteria to the subcritical water-treated solution in order to develop a novel vinegar containing higher concentrations of functional ingredients than the vinegar produced by traditional brewing.

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Compliance with ethical standards

Conflict of interest The authors declare that they have no conflict of interest.

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