WO Publication No. 2020/110108 describes a process for the separation and purification of magnesium lactate from a fermentation broth, comprising the steps of:

(a) providing a clarified fermentation broth from which insoluble impurities have been removed comprising magnesium lactate in a soluble form being the result of a fermentation process, the fermentation broth being at a temperature of about 45°C to about 75°C;

(b) concentrating the clarified broth from step (a) to a concentration of about 150 g/L to about 220 g/L of lactate;

(c) performing at least one cooling crystallization of the concentrated clarified broth from step (b) to obtain magnesium lactate crystals; and

(d) collecting the magnesium lactate crystals obtained.

There remains an unmet need for simple, cost-effective methods with high recovery yields for the crystallization of high purity magnesium L-lactate salt from decomposed organic waste.

SUMMARY

The present disclosure provides a process for preparing high-purity magnesium L-lactate from fermentation broths or other organic waste decomposition products containing lactic acid and/or lactate salts. The process of the present disclosure comprises evaporative crystallization which can utilize fermentation broths or other organic waste decomposition products from diverse origins thereby rendering the process both economically and environmentally beneficial. The process disclosed herein is suitable for fed batch production as well as continuous production in lactic acid recycling facilities.

The disclosure is based, in part, on the unexpected finding that high-purity magnesium L-lactate crystals can be obtained from dispersions of decomposed organic waste such as lactic acid fermentation broths without the necessity of removing D-lactic acid during and/or after fermentation. The process involves the use of evaporative crystallization under specific conditions which yields high-purity magnesium L-lactate crystals already after a single pass despite the presence of significant amounts of impurities in the decomposed waste. Surprisingly, the process of the present disclosure resulted in improved enantiomeric separation even when using decomposed organic

waste containing endogenous D-lactic acid of up to 10wt.%. The process of the present disclosure is therefore simple and economic providing high purity magnesium L-lactate crystals even when a non-homogeneous feedstock comprising high concentrations of soluble and insoluble impurities from municipal, industrial, and commercial origins is used.

The present disclosure provides a process for the formation of high-purity magnesium L-lactate crystals from decomposed organic waste, the process comprising the steps of:

a. providing a clarified dispersion of decomposed organic waste comprising a lactate salt in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified dispersion of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified dispersion of step (a) or the concentrated clarified dispersion of step (b) to obtain a suspension comprising seed magnesium L-lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 90°C and the application of a vacuum to a pressure of about 80 to about 300 mbar, including each value within the specified ranges.

In one aspect, the present disclosure provides a process for the formation of high-purity magnesium L-lactate crystals from decomposed organic waste, the process comprising the steps of:

a. providing a clarified dispersion of decomposed organic waste comprising a lactate salt in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified dispersion of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified dispersion of step (a) or the concentrated clarified dispersion of step (b) to obtain a suspension comprising seed magnesium L-lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 90°C and the application of a vacuum to a pressure of about 80 to about 300 mbar.

According to one example, the decomposed organic waste is obtained from a lactic acid fermentation process. According to another example, the decomposed organic waste is obtained from a lactic acid-containing waste. According to yet another example, the decomposed organic waste is obtained from hydrolysis of polylactic acid polymer.

The present disclosure provides a process for the formation of high-purity magnesium L-lactate crystals from a fermentation broth, the process comprising the steps of:

a. providing a clarified fermentation broth comprising a lactate salt in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified broth of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified fermentation broth of step (a) or the concentrated fermentation broth of step (b) to obtain a suspension comprising seed magnesium L-lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 90°C and the application of a vacuum to a pressure of about 80 to about 300 mbar, including each value within the specified ranges.

The present disclosure provides a process for the formation of high-purity magnesium L-lactate crystals from decomposed organic waste, the process comprising the steps of:

a. providing a clarified dispersion of decomposed organic waste comprising a lactate salt in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified dispersion of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified dispersion of step (a) or the concentrated clarified dispersion of step (b) to obtain a suspension comprising seed magnesium L-lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at a temperature of about 100°C in the absence of a vacuum.

According to some examples, the clarified dispersion or clarified broth comprises decomposed organic waste or fermentation broth from which impurities have been removed using at least one of filtration, centrifugation, flotation, sedimentation, coagulation, flocculation, and decantation. Each possibility represents a separate example. According to additional examples, the clarified dispersion or clarified

broth comprise decomposed organic waste or fermentation broth from which impurities have been removed using filtration and/or centrifugation.

According to other examples, the organic waste comprises a carbohydrate source. According to further examples, the organic waste is selected from food waste, municipal food waste, residential food waste, agricultural waste, industrial food waste from food processing facilities, commercial food waste (from hospitals, restaurants, shopping centers, airports etc.), and a mixture or combination thereof. Each possibility represents a separate example.

According to particular examples, the decomposed organic waste comprises endogenous D-lactic acid. According to specific examples, the decomposed organic waste comprises up to 10wt.% endogenous D-lactic acid.

According to some examples, step (b) is performed to a lactate salt concentration of about 100 to about 130 g/L, including each value within the specified range.

According to various examples, the mixing in step (c) is performed at a speed of about 50 to about 300 rounds per minute (RPM), including each value within the specified range.

According to certain examples, the mixing in step (c) is performed for at least 1 hour. According to other examples, the mixing in step (c) is performed for about 1 to about 6 hours, including each value within the specified range.

According to further examples, steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 80°C, including each value within the specified range. According to other examples, steps (b) through (d) are performed with the application of a vacuum to a pressure of about 150 to about 250 mbar, including each value within the specified range. According to further examples, steps (b) through (d) are performed with the application of a vacuum to a pressure of about 250 to about 350 mbar, including each value within the specified range.

According to particular examples, removing from about 70% to about 90% of water from the suspension in step (d) is performed at an evaporation rate of about 2 to about 5 wt% per hour, including each value within the specified range.

According to various examples, step (e) comprises filtration and/or centrifugation. According to specific examples, step (e) is performed at room temperatures.

According to certain examples, the process further comprises step (f) of washing the obtained magnesium L-lactate crystals. According to particular examples, washing of the obtained magnesium L-lactate crystals is performed in a solvent selected from water, ethanol, propanol, isobutanol, cyclohexane, acetone, ethyl acetate, and a mixture or combination thereof. Each possibility represents a separate example.

According to other examples, the process further comprises step (g) of drying the magnesium L-lactate crystals to a Loss on Drying (LOD) % of about 10% to about 20%, including each value within the specified range. According to particular examples, step (g) is performed at elevated temperatures of about 50°C to about 120°C, including each value within the specified range.

According to additional examples, the obtained magnesium L-lactate crystals are solubilized and re-crystalized by reiterating steps (c) to (e). According to these examples, steps (c) to (e) are performed in cycles, for example between two and six cycles, including each value within the specified range.

According to further examples, the recovery of magnesium L-lactate crystals is at least 90%.

According to other examples, the obtained magnesium L-lactate crystals are characterized by a median size which is smaller than 75 μm. According to certain examples, the obtained magnesium L-lactate crystals are characterized by a size distribution comprising a median size in the range of about 20 to about 100 μm, including each value within the specified range. According to yet other examples, the obtained magnesium L-lactate crystals are characterized by a median size which is larger than 75 μm. According to additional examples, the obtained magnesium L-lactate crystals are characterized by a size distribution comprising a median size in the range of about 100 to about 300 μm, including each value within the specified range.

According to some examples, the obtained magnesium L-lactate crystals comprise less than 3% magnesium D-lactate. According to certain examples, the obtained magnesium L-lactate crystals comprise less than 2% magnesium D-lactate.

According to other examples, the obtained magnesium L-lactate crystals comprise less than 1.5% magnesium D-lactate. According to yet other examples, the obtained magnesium L-lactate crystals comprise less than 1% magnesium D-lactate.

The present disclosure further provides high-purity magnesium L-lactate crystals obtainable by the process disclosed herein. In another aspect, the present disclosure provides a plurality of magnesium L-lactate crystals obtainable by the process disclosed herein.

The present disclosure provides a process for enriching L-lactate enantiomer from an enantiomeric mixture derived from decomposed organic waste, the process comprising the steps of:

a. providing a clarified dispersion of decomposed organic waste comprising a lactate salt comprising an enantiomeric mixture of D- and L-lactate in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified dispersion of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified dispersion of step (a) or the concentrated clarified dispersion of step (b) to obtain a suspension comprising seed magnesium lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals with enriched enantiomeric purity; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 90°C and the application of a vacuum to a pressure of about 80 to about 300 mbar, including each value within the specified ranges.

In another aspect, the present disclosure provides a process for enriching L-lactate enantiomer from an enantiomeric mixture derived from decomposed organic waste, the process comprising the steps of:

a. providing a clarified dispersion of decomposed organic waste comprising a lactate salt comprising an enantiomeric mixture of D- and L-lactate in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified dispersion of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified dispersion of step (a) or the concentrated clarified dispersion of step (b) to obtain a suspension comprising seed magnesium lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals with enriched enantiomeric purity; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 90°C and the application of a vacuum to a pressure of about 80 to about 300 mbar.

In one example, the process provides enrichment of L-lactate enantiomer by 1% or more. In another example, the process provides enrichment of L-lactate enantiomer by 5% or more. In yet another example, the process provides enrichment of L-lactate enantiomer by 10% or more. In particular examples, the process provides enrichment of L-lactate enantiomer of up to 15%. In other examples, the process provides enrichment of L-lactate enantiomer of up to 20%. In yet other examples, the process provides enrichment of L-lactate enantiomer of up to 25%.

It is to be understood that any combination of each of the aspects and the examples disclosed herein is explicitly encompassed within the disclosure of the present disclosure.

Further examples and the full scope of applicability of the present disclosure will become apparent from the detailed description given hereinafter. However, it should be understood that the detailed description and specific examples, while indicating preferred examples of the disclosure, are given by way of illustration only, since various changes and modifications within the spirit and scope of the disclosure will become apparent to those skilled in the art from this detailed description.

BRIEF DESCRIPTION OF THE FIGURES

Figure 1 shows the size distribution of magnesium L-lactate crystals obtained according to some embodiments of the present invention.

Figures 2A-2F show light microscopy images of magnesium L-lactate crystals obtained according to some embodiments of the present invention that remained on top of sieves having cutoff sizes of 710 µm (2A), 500 µm (2B), 300 µm (2C), 100 µm (2D), and 75 µm (2E). Figure 2F shows a light microscopy image of magnesium L-lactate crystals that passed the 75 µm sieve.

Figure 3 shows the size distribution of magnesium L-lactate crystals obtained using cooling crystallization.

Figures 4A-4F show light microscopy images of magnesium L-lactate crystals obtained using cooling crystallization that remained on top of sieves having cutoff sizes of 710 µm (4A), 500 µm (4B), 300 µm (4C), 100 µm (4D), and 75 µm (4E). Figure 4F shows a light microscopy image of magnesium L-lactate crystals that passed the 75 µm sieve.

Figure 5 shows the size distribution of magnesium L-lactate crystals obtained according to some embodiments of the present invention following a recrystallization.

Figure 6 shows the size distribution of magnesium L-lactate crystals obtained using different evaporation rates.

Figure 7 shows the size distribution of magnesium L-lactate crystals obtained according to some embodiments of the present invention from a fed batch constant volume crystallization.

Figure 8 shows a light microscopy image of magnesium L-lactate crystals obtained according to some embodiments of the present invention from a fed batch constant volume crystallization.

CLAIMS

1. A process for the formation of high-purity magnesium L-lactate crystals from decomposed organic waste, the process comprising the steps of:

a. providing a clarified dispersion of decomposed organic waste comprising a lactate salt in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified dispersion of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified dispersion of step (a) or the concentrated clarified dispersion of step (b) to obtain a suspension comprising seed magnesium L-lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 90°C and the application of a vacuum to a pressure of about 80 to about 300 mbar.

2. The process according to claim 1, wherein the clarified dispersion of decomposed organic waste comprises decomposed organic waste from which impurities have been removed using at least one of filtration, centrifugation, flotation, sedimentation, coagulation, flocculation, and decantation; or wherein the organic waste is selected from food waste, municipal food waste, residential food waste, agricultural waste, industrial food waste from food processing facilities, commercial food waste, and a mixture or combination thereof.

3. The process according to claim 1, wherein the decomposed organic waste comprises a fermentation broth.

4. The process according to claim 3, wherein the fermentation broth is obtained from a fermentation process of a carbohydrate source or wherein the fermentation broth is obtained from a fermentation process of an organic waste feedstock.

5. The process according to any one of claims 1 to 4, wherein step (b) is performed to a lactate salt concentration of about 100 to about 130 g/L.

6. The process according to any one of claims 1 to 5, wherein the mixing in step (c) is performed at a speed of about 50 to about 300 rounds per minute (RPM) or wherein the mixing in step (c) is performed for at least 1 hour.

7. The process according to any one of claims 1 to 6, wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 80°C; or wherein steps (b) through (d) are performed with the application of a vacuum to a pressure of about 150 to about 250 mbar.

8. The process according to any one of claims 1 to 7, wherein step (d) is performed at an evaporation rate of about 2 to about 5 wt% per hour.

9. The process according to any one of claims 1 to 8, wherein step (e) comprises filtration and/or centrifugation; or wherein step (e) is performed at room temperatures.

10. The process according to any one of claims 1 to 9, further comprising step (f) of washing the obtained magnesium L-lactate crystals.

11. The process according to claim 10, wherein washing of the obtained magnesium L-lactate crystals is performed in a solvent selected from water, ethanol, propanol, isobutanol, cyclohexane, acetone, ethyl acetate, and a mixture or combination thereof.

12. The process according to any one of claims 1 to 11, further comprising step (g) of drying the magnesium L-lactate crystals to a Loss on Drying (LOD) % of about 10% to about 20%, wherein drying is performed at elevated temperatures of about 50°C to about 120°C.

13. The process according to any one of claims 1 to 12, wherein the obtained magnesium L-lactate crystals are solubilized and re-crystalized by reiterating steps (c) to (e).

14. The process according to any one of claims 1 to 13, wherein the recovery of magnesium L-lactate crystals is at least 90%.

15. The process according to any one of claims 1 to 14, wherein the obtained magnesium L-lactate crystals are characterized by a median size smaller than 75 µm.

16. The process according to any one of claims 1 to 14, wherein the obtained magnesium L-lactate crystals are characterized by a median size larger than 75 µm; or wherein the obtained magnesium L-lactate crystals are characterized by a size distribution comprising a median size in the range of about 100 to about 300 µm.

17. The process according to any one of claims 1 to 16, wherein the obtained magnesium L-lactate crystals comprise less than 3% magnesium D-lactate.

18. A plurality of magnesium L-lactate crystals obtainable by the process of any one of claims 1 to 17.

19. A process for enriching L-lactate enantiomer from an enantiomeric mixture derived from decomposed organic waste, the process comprising the steps of:

a. providing a clarified dispersion of decomposed organic waste comprising a lactate salt comprising an enantiomeric mixture of D- and L-lactate in a concentration of about 50 to about 110 g/L;

b. optionally concentrating the clarified dispersion of step (a) to a lactate salt concentration of about 100 to about 150 g/L;

c. mixing the clarified dispersion of step (a) or the concentrated clarified dispersion of step (b) to obtain a suspension comprising seed magnesium lactate crystals;

d. removing from about 70% to about 90% of water from the suspension of step (c) to obtain magnesium L-lactate crystals with enriched enantiomeric purity; and

e. collecting the magnesium L-lactate crystals obtained in step (d),

wherein steps (b) through (d) are performed at elevated temperature in the range of about 50°C to about 90°C and the application of a vacuum to a pressure of about 80 to about 300 mbar.

20. The process according to claim 19, wherein the obtained magnesium L-lactate crystals comprise less than 1% magnesium D-lactate.