

5 HYDROLYSIS OF USED IONIC LIQUID CATALYST FOR DISPOSAL

TECHNICAL FIELD

This application is directed to a process and apparatus for preparing a used ionic liquid catalyst for disposal.

10 BACKGROUND

Ionic liquid catalysts need to be safely disposed of after use. Without treatment, they can be highly water reactive and unsafe to handle or dispose of.

SUMMARY

15 This application provides a process and apparatus for preparing a used catalyst for disposal, comprising:

 a. hydrolyzing a used ionic liquid catalyst comprising an anhydrous metal halide to produce a hydrolyzed product; and

 b. separating the hydrolyzed product into a liquid phase and a solid phase;

20 wherein the liquid phase comprises a non-water-reactive aqueous phase and a hydrocarbon phase; and wherein the solid phase comprises a solid portion of the hydrolyzed product, that is not water reactive. The apparatus comprises a vessel used to hydrolyze the used ionic liquid catalyst and a separator.

25 This application provides a process for preparing a used catalyst for safe disposal, comprising:

 a. hydrolyzing a used ionic liquid catalyst comprising an anhydrous metal halide with a basic solution to produce a hydrolyzed product wherein a hydrogen halide gas is evolved during the hydrolyzing and the hydrogen halide gas is dissolved into the basic solution to neutralize the hydrogen halide gas;

30 b. separating the hydrolyzed product into a liquid phase and a solid phase; wherein the liquid phase comprises a non-water-reactive aqueous phase having a pH of 4 to 10 and a hydrocarbon phase; and wherein the solid phase is not water reactive.

This application provides an apparatus when used to prepare a used catalyst for safe disposal, comprising:

35 a. a vessel, holding a basic solution, used to hydrolyze a used ionic liquid catalyst comprising an anhydrous metal halide, to produce a hydrolyzed product, wherein a hydrogen halide gas is evolved and dissolved into the basic solution in the vessel to neutralize the hydrogen halide gas; and

40 b. a separator used to separate a liquid phase and a solid phase from the hydrolyzed product; wherein the liquid phase comprises a non-water-reactive aqueous phase having a pH of 4 to 10 and a hydrocarbon phase; and wherein the solid phase is not water reactive.

BRIEF DESCRIPTION OF THE DRAWINGS

5 FIG 1 is a diagram of a flow chart with one embodiment for hydrolysis of ionic liquid catalyst.

DETAILED DESCRIPTION

10 Anhydrous metal-halide-containing used ionic liquid catalyst is treated for safe and economic disposal by hydrolyzing the used ionic liquid catalyst followed by separation which produces a non-water-reactive aqueous phase, a hydrocarbon phase and a solid phase.

15 Prior to the treatment by the process the used catalyst is water reactive and unsuitable for disposal by usual methods. "Water reactive" means that the composition will violently react with moisture, sometimes leading to release of toxic gases, explosions, or fire. Water reactive substances are dangerous when wet because they undergo a chemical reaction with water. This reaction can release a gas that presents a toxic health hazard. In addition, the heat

5 IT IS CLAIMED:

1. A process for preparing a used catalyst for disposal, comprising:
 - a. hydrolyzing a used ionic liquid catalyst comprising an anhydrous metal halide to produce a hydrolyzed product; and
 - b. separating the hydrolyzed product into a liquid phase and a solid phase; wherein
10 the liquid phase comprises a non-water-reactive aqueous phase and a hydrocarbon phase; and wherein the solid phase comprises a solid portion of the hydrolyzed product, that is not water reactive.
2. The process of claim 1, wherein the used ionic liquid catalyst is selected from the group consisting of a hydrocarbyl-substituted-pyridinium chloroaluminate, a hydrocarbyl-
15 substituted-imidazolium chloroaluminate, a quaternary amine chloroaluminate, a trialkyl amine hydrogen chloride chloroaluminate, an alkyl pyridine hydrogen chloride chloroaluminate, and mixtures thereof.
3. The process of claim 1, wherein the anhydrous metal halide is selected from the group consisting of AlCl_3 , AlBr_3 , GaCl_3 , GaBr_3 , InCl_3 , InBr_3 , and mixtures thereof.
- 20 4. The process of claim 1, wherein the hydrolyzing is done with a basic solution comprising a base selected from the group consisting of LiOH , NaOH , KOH , CsOH , RbOH , $\text{Mg}(\text{OH})_2$, $\text{Ca}(\text{OH})_2$, $\text{Sr}(\text{OH})_2$, NH_4OH , $\text{Ba}(\text{OH})_2$, and mixtures thereof.
5. The process of claim 1, wherein less than a full charge of the used ionic liquid catalyst is removed from a hydrocarbon conversion process unit such that the hydrocarbon
25 conversion process unit operates continuously.
6. The process of claim 1, wherein the used ionic liquid catalyst comprises conjunct polymer.
7. The process of claim 1, additionally comprising separating the non-water-reactive aqueous phase from the hydrocarbon phase.
- 30 8. The process of claim 1, wherein a hydrogen halide gas is evolved during the hydrolyzing and the hydrogen halide gas is dissolved into a basic solution used for the hydrolyzing and is neutralized.
9. The process of claim 1, wherein the hydrolyzing proceeds continuously by adding the used ionic liquid catalyst to a hydrolysis vessel while the hydrolyzed product is taken out of the
35 hydrolysis vessel.
10. The process of claim 1, wherein the used ionic liquid catalyst comprises one or more corrosion metals and the one or more corrosion metals, or products thereof, are collected in the solid phase.

- 5 11. The process of claim 1, wherein greater than 80 wt% of the anhydrous metal halide is hydrolyzed and collected in the solid phase.
12. An apparatus for preparing a used catalyst for disposal, comprising:
- 10 a. a vessel used to hydrolyze a used ionic liquid catalyst comprising an anhydrous metal halide, to produce a hydrolyzed product; and
- b. a separator used to separate a liquid phase and a solid phase from the hydrolyzed product; wherein the liquid phase comprises a non-water-reactive aqueous phase and a hydrocarbon phase; and wherein the solid phase comprises a solid portion of the hydrolyzed product, that is not water reactive.
13. The apparatus of claim 12, wherein the vessel used to hydrolyze holds a basic solution
- 15 comprising a base selected from the group consisting of LiOH, NaOH, KOH, CsOH, RbOH, Mg(OH)₂, Ca(OH)₂, Sr(OH)₂, NH₄OH, Ba(OH)₂, and mixtures thereof.
14. The apparatus of claim 12, additionally comprising a liquid/liquid separator used to separate the liquid phase into the non-water-reactive aqueous phase from the hydrocarbon phase.
- 20 15. The apparatus of claim 14, wherein the liquid/liquid separator is a dropping funnel, a separatory funnel, or a centrifuge.

AMENDED CLAIMS

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1. A process for preparing a used catalyst for disposal, comprising:
 - a. hydrolyzing a used ionic liquid catalyst comprising an anhydrous metal halide to produce a hydrolyzed product, wherein a hydrogen halide gas is evolved during the hydrolyzing and the hydrogen halide gas is dissolved into a basic solution used for the hydrolyzing and the hydrogen halide gas is neutralized; and
 - b. separating the hydrolyzed product into a liquid phase and a solid phase; wherein the liquid phase comprises a non-water-reactive aqueous phase having a pH of 4 to 10 and a hydrocarbon phase; and wherein the solid phase is not water reactive.
2. The process of claim 1, wherein the used ionic liquid catalyst is selected from the group consisting of a hydrocarbyl-substituted-pyridinium chloroaluminate, a hydrocarbyl-substituted-imidazolium chloroaluminate, a quaternary amine chloroaluminate, a trialkyl amine hydrogen chloride chloroaluminate, an alkyl pyridine hydrogen chloride chloroaluminate, and mixtures thereof.
3. The process of claim 1, wherein the anhydrous metal halide is selected from the group consisting of AlCl_3 , AlBr_3 , GaCl_3 , GaBr_3 , InCl_3 , InBr_3 , and mixtures thereof.
4. The process of claim 1, wherein the hydrolyzing is done with a basic solution comprising a base selected from the group consisting of LiOH , NaOH , KOH , CsOH , RbOH , $\text{Mg}(\text{OH})_2$, $\text{Ca}(\text{OH})_2$, $\text{Sr}(\text{OH})_2$, NH_4OH , $\text{Ba}(\text{OH})_2$, and mixtures thereof.
5. The process of claim 1, wherein less than a full charge of the used ionic liquid catalyst is removed from a hydrocarbon conversion process unit such that the hydrocarbon conversion process unit operates continuously.

6. The process of claim 1, wherein the used ionic liquid catalyst comprises conjunct polymer.
7. The process of claim 1, additionally comprising separating the non-water-reactive aqueous phase from the hydrocarbon phase.
8. (Cancelled)
9. The process of claim 1, wherein the hydrolyzing proceeds continuously by adding the used ionic liquid catalyst to a hydrolysis vessel while the hydrolyzed product is taken out of the hydrolysis vessel.
10. The process of claim 1, wherein the used ionic liquid catalyst comprises one or more corrosion metals other than aluminum and the one or more corrosion metals, or products thereof, are collected in the solid phase.
11. The process of claim 1, wherein greater than 80 wt% of the anhydrous metal halide is hydrolyzed and collected in the solid phase.
12. An apparatus for preparing a used catalyst for disposal, comprising:
 - a. a vessel, holding a basic solution, used to hydrolyze a used ionic liquid catalyst comprising an anhydrous metal halide, to produce a hydrolyzed product, wherein a hydrogen halide gas is evolved and dissolved into the basic solution in the vessel; and
 - b. a separator used to separate a liquid phase and a solid phase from the hydrolyzed product; wherein the liquid phase comprises a non-water-reactive aqueous phase having a pH of 4 to 10 and a hydrocarbon phase; and wherein the solid phase is not water reactive.
13. The apparatus of claim 12, wherein the basic solution comprises a base selected from the group consisting of LiOH, NaOH, KOH, CsOH, RbOH, Mg(OH)₂, Ca(OH)₂, Sr(OH)₂, NH₄OH, Ba(OH)₂, and mixtures thereof.

14. The apparatus of claim 12, additionally comprising a liquid/liquid separator used to separate the liquid phase into the non-water-reactive aqueous phase from the hydrocarbon phase.
15. The apparatus of claim 14, wherein the liquid/liquid separator is a dropping funnel, a separatory funnel, or a centrifuge.
16. The apparatus of claim 12, wherein the used ionic liquid catalyst comprises one or more corrosion metals other than aluminum and the one or more corrosion metals, or products thereof, are collected in the solid phase.

IT IS CLAIMED:

1. A process for preparing a used catalyst for safe disposal, comprising:
 - a. hydrolyzing a used ionic liquid catalyst comprising an anhydrous metal halide with a basic solution to produce a hydrolyzed product wherein a hydrogen halide gas is evolved during the hydrolyzing and the hydrogen halide gas is dissolved into the basic solution to neutralize the hydrogen halide gas;
 - b. separating the hydrolyzed product into a liquid phase and a solid phase; wherein the liquid phase comprises a non-water-reactive aqueous phase having a pH of 4 to 10 and a hydrocarbon phase; and wherein the solid phase is not water reactive.
2. The process of claim 1, wherein the used ionic liquid catalyst is selected from the group consisting of a hydrocarbyl-substituted-pyridinium chloroaluminate, a hydrocarbyl-substituted-imidazolium chloroaluminate, a quaternary amine chloroaluminate, a trialkyl amine hydrogen chloride chloroaluminate, an alkyl pyridine hydrogen chloride chloroaluminate, and mixtures thereof.
3. The process of claim 1 or 2, wherein the anhydrous metal halide is selected from the group consisting of AlCl_3 , AlBr_3 , GaCl_3 , GaBr_3 , InCl_3 , InBr_3 , and mixtures thereof.
4. The process of any one of the preceding claims, wherein the hydrolyzing is done with a basic solution comprising a base selected from the group consisting of LiOH , NaOH , KOH , CsOH , RbOH , $\text{Mg}(\text{OH})_2$, $\text{Ca}(\text{OH})_2$, $\text{Sr}(\text{OH})_2$, NH_4OH , $\text{Ba}(\text{OH})_2$, and mixtures thereof.
5. The process of any one of the preceding claims, wherein less than a full charge of the used ionic liquid catalyst is removed from a hydrocarbon conversion process unit such that the hydrocarbon conversion process unit operates continuously.
6. The process of any one of the preceding claims, wherein the used ionic liquid catalyst comprises conjunct polymer.
7. The process of any one of the preceding claims, additionally comprising separating the non-water-reactive aqueous phase from the hydrocarbon phase.
8. The process of any one of the preceding claims, wherein the hydrolyzing proceeds continuously by adding the used ionic liquid catalyst to a hydrolysis vessel while the hydrolyzed product is taken out of the hydrolysis vessel.
9. The process of any one of the preceding claims, wherein the used ionic liquid catalyst comprises one or more corrosion metals selected from the group consisting of Co, Cr, Cu, Fe, Mn, Mo, Nb, Ni, Ti, V, and W and the one or more corrosion metals, or products thereof, are collected in the solid phase.

10. The process of any one of the preceding claims, wherein greater than 80 wt% of the anhydrous metal halide is hydrolyzed and collected in the solid phase.
11. The process of any one of the preceding claims, wherein the used ionic liquid catalyst and the basic solution are mixed together at a molar ratio of used ionic liquid catalyst to a base of 0.5:1 to 1:20.
12. The process of any one of the preceding claims, wherein a pH of the basic solution is adjusted to reach a target pH for disposal.
13. The process of any one of the preceding claims, wherein the target pH for disposal enables the non-water-reactive aqueous phase to be sent to a non-hazardous effluent waste handling facility.
14. The process of any one of the preceding claims, wherein the pH is 5 to 9.
15. The process of any one of the preceding claims, wherein the solid phase contains solid precipitates comprising hydrated aluminum chloride.
16. The process of any one of the preceding claims, wherein the hydrolyzing is performed with control of a hydrolysis temperature.
17. An apparatus when used to prepare a used catalyst for safe disposal, comprising:
 - a. a vessel, holding a basic solution, used to hydrolyze a used ionic liquid catalyst comprising an anhydrous metal halide, to produce a hydrolyzed product, wherein a hydrogen halide gas is evolved and dissolved into the basic solution in the vessel to neutralize the hydrogen halide gas; and
 - b. a separator used to separate a liquid phase and a solid phase from the hydrolyzed product; wherein the liquid phase comprises a non-water-reactive aqueous phase having a pH of 4 to 10 and a hydrocarbon phase; and wherein the solid phase is not water reactive.
- 25 18. The apparatus of claim 17, wherein the vessel used to hydrolyze holds a basic solution comprising a base selected from the group consisting of LiOH, NaOH, KOH, CsOH, RbOH, Mg(OH)₂, Ca(OH)₂, Sr(OH)₂, NH₄OH, Ba(OH)₂, and mixtures thereof.
19. The apparatus of claim 17 or 18, additionally comprising a liquid/liquid separator used to separate the liquid phase into the non-water-reactive aqueous phase from the hydrocarbon phase.
- 30 20. The apparatus of claim 19, wherein the liquid/liquid separator is a dropping funnel, a separatory funnel, or a centrifuge.