Publication No. 20100129921 (Timken *et al.*), the disclosure of which is incorporated by reference herein in its entirety.

As noted above, sample preparation for wet chemistry analyses is both time consuming and may consume significant quantities of the ionic liquid. Accordingly, there is a need for a non-destructive, convenient, and efficient method for monitoring the composition of an ionic liquid before or during an ionic liquid catalyzed process.

SUMMARY

Due to possible variation in the composition and catalytic activity of ionic liquids, it may be valuable to monitor the catalytic activity of an ionic liquid catalyst during or prior to a process using the ionic liquid catalyst. For example, during chloroaluminate ionic liquid catalyzed hydrocarbon conversion reactions, byproduct hydrocarbons known as conjunct polymer react with and deactivate the ionic liquid.

According to a first aspect, there is provided a method for monitoring an ionic liquid, wherein the method comprises monitoring the ionic liquid during a hydrocarbon conversion process catalyzed by the ionic liquid wherein a conjunct polymer is formed in the ionic liquid during the hydrocarbon conversion process, comprising: contacting an infrared (IR) transmissive medium with the ionic liquid; recording an IR spectrum of the ionic liquid; and determining a concentration of the conjunct polymer in the ionic liquid based on the IR spectrum.

According to a second aspect, there is provided a method for monitoring an ionic liquid, wherein the method comprises monitoring the ionic liquid during a hydrocarbon conversion process catalyzed by the ionic liquid wherein a conjunct polymer is formed in the ionic liquid during the hydrocarbon conversion process, comprising: contacting an infrared (IR) transmissive medium with the ionic liquid; recording an IR spectrum of the ionic liquid; and quantitatively determining a concentration of the conjunct polymer in the ionic liquid based on the IR spectrum, using solely an IR band around 1370 cm^{-1}.

According to a third aspect, there is provided a method for controlling an ionic liquid catalyzed hydrocarbon conversion process in which the ionic liquid is at least partially deactivated during the process to form used catalyst, wherein the process includes regenerating the used catalyst under catalyst regeneration conditions in a catalyst regeneration zone to provide regenerated catalyst, and an IR spectral data indicates an extent of catalyst regeneration, comprising: recording infrared (IR) spectral data of the ionic liquid during the process; determining an amount of a conjunct polymer in the ionic liquid based on the IR spectral data; and changing at least one of the catalyst regeneration conditions based on the IR spectral data.

According to a fourth aspect, there is provided a method for controlling an ionic liquid catalyzed hydrocarbon conversion process in which the ionic liquid is at least partially deactivated during the process to form a used catalyst, wherein the process includes regenerating the used catalyst under catalyst regeneration conditions in a catalyst regeneration zone to provide a regenerated catalyst, and an IR spectral data indicates an extent of catalyst regeneration, comprising: recording infrared (IR) spectral data of an ionic liquid during the process; quantitatively determining an amount of a conjunct polymer in the ionic liquid based on the IR spectral data, using solely an IR band around 1370 cm^{-1}; and changing at least one of the catalyst regeneration conditions based on the IR spectral data.

Accordingly, in an embodiment of the instant invention the catalytic activity of the ionic liquid may be monitored in order to facilitate the control of ionic liquid catalyzed processes.

According to an ~~aspect~~embodiment of the instant invention, there is provided a method for quantifying one or more chemical characteristics of a fresh, used, or regenerated ionic liquid catalyst before or during an ionic liquid catalyzed process, wherein the method can rapidly provide quantitative data for a range of analytes from a single test with little or no sample preparation. In an embodiment, analysis of ionic liquid may be performed online during ionic liquid catalyzed processes, thereby eliminating the need for sampling *per se*.

According to one embodiment of the instant invention, a method for assessing the purity of a batch of an ionic liquid comprises contacting an infrared (IR) transmissive medium with the ionic liquid, recording an IR spectrum of the ionic liquid, and determining a purity level of the ionic liquid based on the IR spectrum.

WHAT IS CLAIMED IS:

1. A method for monitoring an ionic liquid, comprising:
contacting an infrared (IR) transmissive medium with the ionic liquid;
recording an IR spectrum of the ionic liquid; and
quantifying at least one chemical characteristic of the ionic liquid based on the IR spectrum.

2. The method according to claim 1, wherein the quantifying step comprises determining a purity level of the ionic liquid.

3. The method according to claim 1, wherein the method comprises monitoring the ionic liquid during a hydrocarbon conversion process catalyzed by the ionic liquid.

4. The method according to claim 3, wherein the IR spectrum is continuously recorded, *in situ*, in attenuated total reflectance (ATR) mode during the hydrocarbon conversion process.

5. The method according to claim 3, wherein the IR spectrum is periodically recorded, *ex situ*, in ATR mode or transmission mode during the hydrocarbon conversion process.

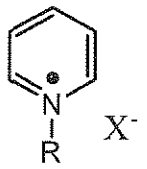
6. The method according to claim 3, wherein conjunct polymer is formed in the ionic liquid during the hydrocarbon conversion process, and the quantifying step comprises determining a concentration of the conjunct polymer in the ionic liquid.

7. The method according to claim 3, wherein the contacting step comprises contacting the ionic liquid with the IR transmissive medium within an in-line ATR-IR unit during the hydrocarbon conversion process.

8. The method according to claim 1, wherein the recording step comprises obtaining a plurality of IR spectra during a defined time period, and the quantifying

step comprises monitoring a change in the at least one chemical characteristic during the defined time period.

9. The method according to claim 1, wherein the ionic liquid comprises at least one compound of the general formulas A and B:



A



B

wherein R is selected from the group consisting of H, methyl, ethyl, propyl, butyl, pentyl or hexyl, each of R_1 and R_2 is selected from the group consisting of H, methyl, ethyl, propyl, butyl, pentyl or hexyl, wherein R_1 and R_2 may or may not be the same, and X is a chloroaluminate.

10. The method according to claim 1, wherein the ionic liquid comprises N-butylpyridinium chloroaluminate.

11. A method for controlling an ionic liquid catalyzed hydrocarbon conversion process, comprising:

recording infrared (IR) spectral data of the ionic liquid during the process; and

determining a condition of the ionic liquid during the process based on the IR spectral data.

12. The method according to claim 11, wherein the method further comprises changing at least one process parameter based on the IR spectral data.

13. The method according to claim 11, wherein the IR spectral data is recorded in ATR mode or transmission mode.

14. The method according to claim 11, wherein the IR spectral data is periodically recorded, *ex situ,* during the process in attenuated total reflectance (ATR) mode or transmission mode.

15. The method according to claim 11, wherein the IR spectral data is continuously recorded, *in situ,* by an in-line ATR-IR unit during the process.

16. The method according to claim 11, wherein the process is performed in a reactor, the IR spectral data is recorded by an in-line ATR-IR unit during the process, and the ATR-IR unit is disposed downstream from a catalyst storage unit and upstream from the reactor.

17. The method according to claim 11, wherein conjunct polymer is formed as a byproduct of the process, the conjunct polymer accumulates in the ionic liquid during the process, and the IR spectral data indicates an amount of the conjunct polymer in the ionic liquid.

18. The method according to claim 11, wherein the ionic liquid is at least partially deactivated during the hydrocarbon conversion process, and the IR spectral data indicates an extent of ionic liquid deactivation.

19. The method according to claim 11, wherein:

the ionic liquid is at least partially deactivated during the process to form used catalyst,

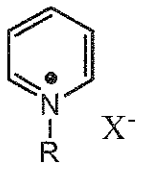
the process includes regenerating the used catalyst under catalyst regeneration conditions in a catalyst regeneration zone to provide regenerated catalyst, and

the IR spectral data indicates an extent of catalyst regeneration.

20. The method according to claim 19, further comprising: changing at least one of the catalyst regeneration conditions based on the IR spectral data.

21. The method according to claim 19, wherein the IR spectral data is recorded by an in-line ATR-IR unit disposed downstream from the catalyst regeneration zone.

22. The method according to claim 11, wherein the process comprises an isoparaffin/olefin alkylation reaction, and the ionic liquid comprises at least one compound of the general formulas A and B:



A



B

wherein R is selected from the group consisting of H, methyl, ethyl, propyl, butyl, pentyl or hexyl, each of R_1 and R_2 is selected from the group consisting of H, methyl, ethyl, propyl, butyl, pentyl or hexyl, wherein R_1 and R_2 may or may not be the same, and X is a chloroaluminate.

23. An ionic liquid catalyzed hydrocarbon conversion process, comprising:

contacting a hydrocarbon feed with fresh ionic liquid in a hydrocarbon conversion zone under hydrocarbon conversion conditions to provide: i) a hydrocarbon phase, and ii) a catalyst phase comprising used ionic liquid;

regenerating at least a portion of the used ionic liquid in a regeneration zone under regeneration conditions to provide regenerated ionic liquid; and

recording infrared (IR) spectral data of at least one of the fresh ionic liquid, the used ionic liquid, and the regenerated ionic liquid, wherein:

the IR spectral data is indicative of catalytic activity of the fresh ionic liquid, the used ionic liquid, and the regenerated ionic liquid, and

the IR spectral data is recorded in attenuated total reflectance (ATR) mode or transmission mode.

24. The process according to claim 23, wherein the hydrocarbon conversion process comprises a chloroaluminate ionic liquid catalyzed alkylation reaction.

25. The process according to claim 24, wherein the hydrocarbon conversion process comprises an isoparaffin/olefin alkylation reaction, and the chloroaluminate ionic liquid comprises N-butylpyridinium chloroaluminate.

WHAT IS CLAIMED IS:

1. A method for monitoring an ionic liquid, wherein the method comprises monitoring the ionic liquid during a hydrocarbon conversion process catalyzed by the ionic liquid wherein a conjunct polymer is formed in the ionic liquid during the hydrocarbon conversion process, comprising:

contacting an infrared (IR) transmissive medium with the ionic liquid;

recording an IR spectrum of the ionic liquid; and

determining a concentration of the conjunct polymer in the ionic liquid based on the IR spectrum.

2. The method according to claim 1, wherein the IR spectrum is continuously recorded, *in situ*, in attenuated total reflectance (ATR) mode during the hydrocarbon conversion process.

3. A method for monitoring an ionic liquid, wherein the method comprises monitoring the ionic liquid during a hydrocarbon conversion process catalyzed by the ionic liquid wherein a conjunct polymer is formed in the ionic liquid during the hydrocarbon conversion process, comprising:

contacting an infrared (IR) transmissive medium with the ionic liquid;

recording an IR spectrum of the ionic liquid; and

quantitatively determining a concentration of the conjunct polymer in the ionic liquid based on the IR spectrum, using solely an IR band around 1370 cm^{-1}.

4. The method according to claims 1 or 3, wherein the IR spectrum is periodically recorded, *ex situ*, in ATR mode or transmission mode during the hydrocarbon conversion process.

5. The method according to claims 1 or 3, wherein the contacting step comprises contacting the ionic liquid with the IR transmissive medium within an in-line ATR-IR unit during the hydrocarbon conversion process.

6. The method according to any one of the preceding claims, wherein the ionic liquid comprises N-butylpyridinium chloroaluminate.

7. A method for controlling an ionic liquid catalyzed hydrocarbon conversion process in which the ionic liquid is at least partially deactivated during the process to form used catalyst, wherein the process includes regenerating the used catalyst under catalyst regeneration conditions in a catalyst regeneration zone to provide regenerated catalyst, and an IR spectral data indicates an extent of catalyst regeneration, comprising:

recording infrared (IR) spectral data of the ionic liquid during the process;

determining an amount of a conjunct polymer in the ionic liquid based on the IR spectral data; and changing at least one of the catalyst regeneration conditions based on the IR spectral data.

8. A method for controlling an ionic liquid catalyzed hydrocarbon conversion process in which the ionic liquid is at least partially deactivated during the process to form a used catalyst, wherein the process includes regenerating the used catalyst under catalyst regeneration conditions in a catalyst regeneration zone to provide a regenerated catalyst, and an IR spectral data indicates an extent of catalyst regeneration, comprising:

recording infrared (IR) spectral data of an ionic liquid during the process;

quantitatively determining an amount of a conjunct polymer in the ionic liquid based on the IR spectral data, using solely an IR band around 1370 cm^{-1}; and changing at least one of the catalyst regeneration conditions based on the IR spectral data.

9. The method according to claim 7 or 8, wherein the IR spectral data is recorded in ATR mode or transmission mode.

10. The method according to claim 7 or 8, wherein the IR spectral data is periodically recorded, *ex situ,* during the process in attenuated total reflectance (ATR) mode or transmission mode.

11. The method according to claim 7 or 8, wherein the IR spectral data is continuously recorded, *in situ,* by an in-line ATR-IR unit during the process.

12. The method according to claim 7 or 8, wherein the process is performed in a reactor, the IR spectral data is recorded by an in-line ATR-IR unit during the process, and the ATR-IR unit is disposed downstream from a catalyst storage unit and upstream from the reactor.

13. The method according to claim 7 or 8, wherein the conjunct polymer is formed as a byproduct of the process, the conjunct polymer accumulates in the ionic liquid during the process, and the IR spectral data indicates an amount of the conjunct polymer in the ionic liquid.

14. The method according to claim 1, wherein the IR band at 1370cm^{-1} is used to determine conjunct polymer concentration.

15. The method according to claim 7, wherein the IR band at 1370 cm^{-1} is used to determine conjunct polymer concentration.

16. The method according to claim 1 or 3, wherein the determining the amount of the conjunct polymer in the ionic liquid does not use an internal standard.

17. The method according to claim 7 or 8, wherein the determining the amount of the conjunct polymer in the ionic liquid does not use an internal standard.

18. The method of claim 1 or 3, wherein the infrared (IR) transmissive medium is selected from the group consisting of silver chloride, zinc selenide, barium fluoride, thallium bromide-thallium iodide, germanium, diamond, and zinc selenide.

19. The method of claim 1 or 3, wherein the recording the IR spectrum of the ionic liquid uses an attenuated total reflectance IR unit and the infrared (IR) transmissive medium is selected from the group consisting of thallium bromide-thallium iodide, germanium, diamond, and zinc selenide.

20. The method of claim 7 or 8, wherein the step of recording infrared (IR) spectral data of the ionic liquid uses an attenuated total reflectance IR unit having an

infrared (IR) transmissive medium selected from the group consisting of thallium bromide-thallium iodide, germanium, diamond, and zinc selenide.