



(43) International Publication Date  
14 January 2016 (14.01.2016)

(10) International Publication Number  
**WO 2016/005845 A2**

- (51) International Patent Classification: Not classified
- (21) International Application Number: PCT/IB2015/054770
- (22) International Filing Date: 25 June 2015 (25.06.2015)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data: 2186/MUM/2014 5 July 2014 (05.07.2014) IN
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- (81) Designated States (*unless otherwise indicated, for every kind of national protection available*): AE, AG, AL, AM,

AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.

- (84) Designated States (*unless otherwise indicated, for every kind of regional protection available*): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

**Declarations under Rule 4.17:**

- *as to the identity of the inventor (Rule 4.17(i))*
- *of inventorship (Rule 4.17(iv))*

**Published:**

- *without international search report and to be republished upon receipt of that report (Rule 48.2(g))*

(54) Title: PROCESSING OF DEACTIVATED IONIC LIQUIDS

(57) Abstract: The present disclosure relates to a process for separating the cationic and anionic components from deactivated ionic liquids. The process includes reacting the deactivated ionic liquid with 8-hydroxyquinoline to precipitate the anionic component, separating the precipitate followed by extracting the deactivating components from the cationic component. The anionic precipitate and the deactivating component free cationic component are subsequently recovered and reused for different applications. The ionic liquid can be reconstituted from the cationic component and another anionic component for use as a fresh ionic liquid.



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## PROCESSING OF DEACTIVATED IONIC LIQUIDS

### FIELD

The present disclosure relates to ionic liquids. Particularly, the present disclosure relates to a process for reconstitution of deactivated ionic liquids and separating its cationic and anionic components.

### BACKGROUND

Ionic compounds, as the name suggests, are compounds comprising cations and anions. Typically, they consist of salts having melting point below 100 °C. Ionic liquids are known to be used in various applications such as catalysts, solvents and electrolytes in processes such as alkylation, polymerization, dimerization, oligomerization, acetylation, metatheses and copolymerization. For instance, United States Patent No. 7432408 recites a method for alkylation of isoparaffin and C<sub>2</sub>-C<sub>5</sub> olefins using ionic liquids such as 1-butyl-4-methyl-pyridinium chloroaluminate (BMP), 1-butyl-pyridinium chloroaluminate (BP), 1-butyl-3-methyl-imidazolium chloroaluminate (BMIM) and 1-H-pyridinium chloroaluminate (HP) as the catalyst. United States Patent No. 7495144 also describes a method for alkylation of isoparaffin and C<sub>2</sub>-C<sub>5</sub> olefins using a composite ionic liquid catalyst, wherein the ionic liquid is a mixture of acid ionic liquids such as 1-butyl-4-methyl-pyridinium chloroaluminate (BMP), 1-butyl-pyridinium chloroaluminate (BP), 1-butyl-3-methyl-imidazolium chloroaluminate (BMIM) and 1-H-pyridinium chloroaluminate (HP) and metal halides such as AlCl<sub>3</sub>.

Ammonium, phosphonium, sulphonium, pyridinium and imidazolium are some of the commonly used cations; whereas BF<sub>4</sub><sup>-</sup>, PF<sub>6</sub><sup>-</sup>, haloaluminates such as Al<sub>2</sub>Cl<sub>7</sub><sup>-</sup> and Al<sub>2</sub>Br<sub>7</sub><sup>-</sup>, [(CF<sub>3</sub>SO<sub>2</sub>)<sub>2</sub>N]<sup>-</sup>, alkyl sulphates (RSO<sub>3</sub><sup>-</sup>), carboxylates (RCO<sub>2</sub><sup>-</sup>) are some of the commonly used anions in ionic liquids. However, when the haloaluminate containing ionic liquids are used in any of the afore-stated reactions, they get

deactivated due to various chemical entities present in the reaction such as hydrocarbons, conjunct polymers and water. Thus, upon the completion of the reaction, the ionic liquids, due to their deactivated state, are incapable of being reused for other reactions. Replenishing the stock becomes imperative; however the expensive nature of the chemical results in an exponential increase in the processing cost. Moreover, as the used ionic liquids have to be discarded, tremendous amount of waste is generated and valuable reagents get wasted.

Attempts have been made to reuse the spent ionic liquids. United States Patent Application No. 20100160145 recites a process for breaking down an ionic liquid catalyst and employs a secondary alcohol to achieve this. WO 2010062902 also describes a process for recycling ionic liquids. However, the process of WO 2010062902 facilitates this by removing aluminum trichloride from the ionic liquid by way of cooling or cooling and seeding the ionic liquid to precipitate out the aluminum trichloride.

The techniques that have been used for reuse of ionic liquids are, however, associated with certain disadvantages such as use of expensive reagents and time-consuming process steps. The present disclosure, therefore, provides a process for the reconstitution of ionic liquids which is cost effective as compared to the conventional process, and also generates certain reaction products that may be effectively harnessed.

## **OBJECTS**

Some of the objects of the present disclosure, of which at least one embodiment is adapted to provide, are described herein below:

It is an object of the present disclosure to provide a process for the reconstitution of deactivated ionic liquids.

It is another object of the present disclosure to provide a process for the separation of cationic and anionic components of the deactivated ionic liquid and for the reconstitution of fresh ionic liquids.

It is yet another object of the present disclosure to provide a process for the reconstitution of fresh ionic liquids from deactivated ionic liquid, which is simple and economical.

It is still another object of the present disclosure to provide a process for the reconstitution of fresh ionic liquids, which is environment friendly.

It is yet another object of the present disclosure to provide a process for the reconstitution of deactivated ionic liquids, which yields reaction products having industrially useful applications.

It is still another object of the present disclosure to ameliorate one or more problems of the conventional processes or to at least provide a useful alternative.

Other objects and advantages of the present disclosure will be more apparent from the following description which is not intended to limit the scope of the present disclosure.

## **SUMMARY**

The present disclosure provides a process for separating the cationic and anionic components from deactivated ionic liquids, said process comprising the following steps:

- a. admixing deactivated ionic liquid in a first solvent and heating at a temperature ranging from 60 to 80 °C to obtain a first admixture;
- b. admixing 8-hydroxyquinoline in a second solvent and heating at a temperature ranging from 60 to 80 °C to obtain a second admixture;
- c. preparing a dispersion by admixing said first admixture with said second admixture, in a drop-wise manner, while maintaining the pH from 4 to 7 by using at least one pH adjusting agent;
- d. cooling said dispersion to a temperature below 60 °C followed by filtering to obtain:

- i. a residue comprising a precipitate of said anionic component and 8-hydroxyquinoline; and
- ii. a filtrate comprising said cationic component, deactivating components of said ionic liquid and said second solvent;
- e. washing said residue with said second solvent followed by drying to obtain purified precipitate;
- f. distilling said filtrate to remove said second solvent and obtain a mass comprising said cationic component and said deactivating components;
- g. extracting said deactivating components from said mass by at least one extracting agent to obtain the cationic component devoid of said deactivating components; and
- h. crystallizing said cationic component by at least one crystallizing agent to recover said cationic component.

Typically, the cationic component is selected from the group consisting of 1-butyl-3-methyl imidazolium bromide, 1-butyl-3-methyl imidazolium chloride, 1-butyl-4-methylpyridinium chloride and 1-butyl-4-methylpyridinium bromide; the anionic component is aluminum chloride and the deactivating component is at least one from the group consisting of polymers, tar, hydrocarbons and moisture. The precipitate is a tris(8-hydroxyquinolato) aluminum (III) complex and is characterized as a fluorescent material for use in organic light-emitting diodes.

## DETAILED DESCRIPTION

The disclosure will now be described with reference to the accompanying embodiments which do not limit the scope and ambit of the disclosure. The description provided is purely by way of example and illustration.

The embodiments herein and the various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the following description. Descriptions of well-known components and processing techniques are omitted so as to not unnecessarily obscure the embodiments herein. The examples used

herein are intended merely to facilitate an understanding of ways in which the embodiments herein may be practiced and to further enable those of skill in the art to practice the embodiments herein. Accordingly, the examples should not be construed as limiting the scope of the embodiments herein.

The description of the specific embodiments will so fully reveal the general nature of the embodiments herein that others can, by applying current knowledge, readily modify and/or adapt for various applications such specific embodiments without departing from the generic concept, and, therefore, such adaptations and modifications should and are intended to be comprehended within the meaning and range of equivalents of the disclosed embodiments. It is to be understood that the phraseology or terminology employed herein is for the purpose of description and not of limitation. Therefore, while the embodiments herein have been described in terms of preferred embodiments, those skilled in the art will recognize that the embodiments herein can be practiced with modification within the spirit and scope of the embodiments as described herein.

Ionic liquids are used as catalysts, solvents and electrolytes in different reactions such as polymerization and alkylation. During these reactions, the ionic liquids get deactivated due to contamination with different chemical entities such as polymers and hydrocarbons. The present disclosure provides a process for the recovery of the anionic component in a complex form and the cationic components of the deactivated ionic liquids that may be recycled and reused for different applications. The ionic liquid can be reconstituted from the cationic component and fresh anionic component for use as a fresh ionic liquid.

The process of the present disclosure achieves the reconstitution of the ionic liquids by effecting the separation of its cationic and anionic components from the deactivating components and reconstituting the ionic liquid by combining the cationic component obtained with fresh anionic component to obtain a freshly reconstituted ionic liquid.

Typically, the cationic component of the present disclosure is selected from the group consisting of 1-butyl-3-methyl imidazolium bromide, 1-butyl-3-methyl imidazolium chloride, 1-butyl-4-methylpyridinium chloride and 1-butyl-4-methylpyridinium bromide. The anionic component is a metal chloride, particularly, aluminum chloride. The deactivating components of the present disclosure are from the group consisting of polymers, tar, hydrocarbons and moisture.

The process initially includes admixing the deactivated ionic liquid in a first solvent and heating it at a temperature ranging from 60 to 80 °C to obtain a first admixture. The first solvent of the present disclosure can be toluene. Next, 8-hydroxyquinoline is admixed in a second solvent and heated at a temperature ranging from 60 to 80 °C to obtain a second admixture. Typically, the second solvent is ethanol.

The first admixture is then incorporated in the second admixture, in a drop wise manner, while maintaining the pH from 4 to 7 to obtain a dispersion. The pH is maintained in the desired range by using at least one pH adjusting agent from the group consisting of sodium acetate, sodium hydroxide and sodium carbonate.

The dispersion is then cooled to a temperature below 60 °C and filtered to obtain a residue and a filtrate. The residue is a precipitate of the aluminum ion present in aluminum chloride and 8-hydroxyquinoline – a result of the step of admixing said first and second admixtures. Typically, the precipitate that results is a tris(8-hydroxyquinolato) aluminum (III) complex. The residue is then washed with the second solvent followed by drying to obtain the purified precipitate, which may be used for different applications.

The filtrate consists of the cationic component, the deactivating components and the second solvent. Upon distillation, the second solvent is removed to leave behind a mass consisting of the cationic component and the deactivating components. The deactivating components are then extracted from the reaction mass to obtain the cationic component devoid of deactivating components. The extracting agent of the

present disclosure is at least one from the group consisting of ethylene acetate and toluene.

The deactivating agent-free cationic component is further crystallized by means of at least one crystallizing agent to recover the cationic component. The crystallizing agent of the present disclosure is at least one from the group consisting of acetonitrile and dichloromethane. Once the deactivating components are separated, the cationic component is ready to be combined with another anionic component for use as a fresh ionic liquid.

Therefore, in the present process the deactivated ionic liquid is reconstituted and can be recycled. The separated anionic component complex obtained according to the present process is of fluorescent nature. It may be further used for different applications such as a source of fluorescence to be fitted in organic light-emitting diodes. Thus, the process of the present disclosure effectively reuses the deactivated ionic liquid which would earlier have been discarded.

The present disclosure is further described in light of the experiments provided herein below which are set forth for illustration purpose only and not to be construed for limiting the scope of the disclosure. These laboratory scale experiments can be scaled up to industrial/commercial scale.

### **Example 1: Process for the separation and recovery of the cationic and anionic components from deactivated and fresh ionic liquids**

#### Preparation of fresh ionic liquid catalyst (1-Butyl-3-methylimidazolium bromide + aluminum chloride)

The setup consisted of a 5 L three necked round bottom (RB) flask fitted with an overhead stirrer and placed in an ice bath at 0-5 °C. The flask was clamped to provide stability under stirring. The whole assembly was kept under nitrogen atmosphere. 680 g of [BMIM]Br was weighed and carefully charged into the flask through a funnel.



Stirring was started at a slow speed. Next, 830 g of  $\text{AlCl}_3$  was weighed and added slowly into the flask. The charging of  $\text{AlCl}_3$  was completed in 1.5 hours after which the mixture was stirred for 2 hours to mix the reactants properly. The final catalyst was closed tightly under nitrogen conditions.

#### Separation and recovery of the cationic and anionic components

0.264 g of the afore-stated fresh ionic liquid catalyst was taken in a beaker to which 5 ml of ethanol was added and to a temperature of 67-68 °C. In a separate conical flask, 0.6 g of 8-hydroxyquinoline and 5 ml of ethanol were taken and heated to a temperature of 67-68 °C. The ionic liquid solution was slowly added to the 8-hydroxyquinoline solution, while maintaining the pH at 5-6 by drop wise addition of saturated sodium acetate solution. The pH was measured by using a pH meter. A yellow color solid of tris-(8-hydroxyquinolato) aluminum(III) precipitated out whereas the BMIMBr got dissolved in ethanol. The dispersion was cooled the precipitate was filtered. The precipitate was further washed with ethanol to remove the remaining BMIMBr and dried to obtain 95% fluorescent tris-(8-hydroxyquinolato) aluminum(III). The solvent phase of the dispersion was distilled to remove ethanol and toluene to separate BMIMBr. BMIMBr was further crystallized using acetonitrile to yield 61% of BMIMBr.

#### Preparation of deactivated ionic liquid catalyst (Alkylation using the ionic liquid catalyst prepared herein above)

9.2 liters of a paraffin stream containing 10-15% of  $\text{C}_{10}$  to  $\text{C}_{14}$  olefins and 3.6 liters of benzene were added into a 25 liter round bottom reactor kept inside a heating mantle. The agitator was started and the heating coils were switched on. When the mixture attained the temperature of 45 °C, 0.13 kg of the fresh catalyst prepared herein above was charged and stirred for 10 minutes. After standing for 10 minutes, the hydrocarbon and the catalyst layers were separated and the bottom catalyst layer was recycled back with the same quantities of a fresh olefin stream and benzene.

### Separation and recovery of the cationic and anionic components

0.264 g of the afore-stated deactivated ionic liquid catalyst was taken in a beaker to which 15 ml of toluene was added and heated to a temperature of 67-68 °C. In a separate conical flask, 0.6 g of 8-hydroxyquinoline and 10 ml of ethanol were taken and heated to a temperature of 67-68 °C. The deactivated ionic liquid catalyst solution prepared herein above was slowly added to the 8-hydroxyquinoline solution, while maintaining the pH at 5-6 by drop wise addition of saturated sodium acetate solution. The pH was measured by using a pH meter. A yellow color solid of tris-(8-hydroxyquinolato) aluminum(III) precipitated out whereas the BMIMBr got dissolved in ethanol. The dispersion was cooled the precipitate was filtered. The precipitate was further washed with ethanol to remove the remaining BMIMBr and dried to obtain 96% fluorescent tris-(8-hydroxyquinolato) aluminum(III). The solvent phase of the dispersion was distilled to remove ethanol and toluene and leave behind a slurry containing BMIMBr and conjunct polymers. These conjunct polymers were removed by extraction with ethyl acetate and BMIMBr was further crystallized using acetonitrile to yield 58% of BMIMBr.

### **Inference:**

The process of the present disclosure for separating the cationic and anionic components from ionic liquids - can be successfully applied for both fresh and deactivated ionic liquids.

### **Example 2: Process for the separation and recovery of the cationic and anionic components from deactivated ionic liquids**

#### Preparation of fresh ionic liquid catalyst (1-Butyl-3-methylimidazolium chloride + aluminum chloride)

The setup consisted of a 5 L three necked round bottom (RB) flask fitted with an overhead stirrer and placed in an ice bath at 0-5 °C. The flask was clamped to provide stability under stirring. The whole assembly was kept under nitrogen atmosphere. 542 g of [BMIM]Cl was weighed and carefully charged into the flask through a funnel.

Stirring was started at a slow speed. Next, 830 g of  $\text{AlCl}_3$  was weighed and added slowly into the flask. The charging of  $\text{AlCl}_3$  was completed in 1.5 hours after which the mixture was stirred for 2 hours to mix the raw materials properly. The final catalyst was closed tightly under nitrogen conditions.

Preparation of deactivated ionic liquid catalyst (Alkylation using the ionic liquid catalyst prepared herein above)

9.2 liters of a paraffin stream containing 10-15% of  $\text{C}_{10}$  to  $\text{C}_{14}$  olefins and 3.6 liters of benzene were added into a 25 liter round bottom reactor kept inside a heating mantle. The agitator was started and the heating coils were switched on. When the mixture attained the temperature of  $45^\circ\text{C}$ , 0.13 kg of the catalyst prepared herein above was charged and stirred for 10 minutes. After standing for 10 minutes, the hydrocarbon and the catalyst layers were separated and the bottom catalyst layer was recycled back with the same quantities of a fresh olefin stream and benzene.

Separation and recovery of the cationic and anionic components

0.264 g of deactivated ionic liquid was taken in a beaker to which 15 ml of toluene was added and heated to a temperature of  $67-68^\circ\text{C}$ . In a separate conical flask, 0.6 g of 8-hydroxyquinoline and add 10 ml of ethanol was taken and heated to a temperature of  $67-68^\circ\text{C}$ . The deactivated ionic liquid catalyst solution prepared herein above was slowly added to the 8-hydroxyquinoline solution, while maintaining the pH at 5-6 by drop wise addition of saturated sodium acetate solution. The pH was measured by using a pH meter. A yellow color solid of tris-(8-hydroxyquinolato) aluminum(III) precipitated out whereas the BMIMCl got dissolved in ethanol. The dispersion was cooled the precipitate was filtered. The precipitate was further washed with ethanol to remove the remaining BMIMCl and dried to obtain 95% fluorescent tris-(8-hydroxyquinolato) aluminum(III). The solvent phase of the dispersion was distilled to remove ethanol and leave behind slurry containing BMIMCl and conjunct polymers. These conjunct polymers are removed by extraction with ethyl acetate and BMIMCl was further crystallized using acetonitrile to yield 62-78% of BMIMCl.

**Inference:**

The process of the present disclosure for separating the cationic and anionic components from ionic liquids can be successfully applied for deactivated ionic liquids.

Throughout this specification the word “comprise”, or variations such as “comprises” or “comprising”, will be understood to imply the inclusion of a stated element, integer or step, or group of elements, integers or steps, but not the exclusion of any other element, integer or step, or group of elements, integers or steps.

The use of the expression “at least” or “at least one” suggests the use of one or more elements or ingredients or quantities, as the use may be in the embodiment of the disclosure to achieve one or more of the desired objects or results.

Any discussion of documents, acts, materials, devices, articles or the like that has been included in this specification is solely for the purpose of providing a context for the disclosure. It is not to be taken as an admission that any or all of these matters form a part of the prior art base or were common general knowledge in the field relevant to the disclosure as it existed anywhere before the priority date of this application.

The numerical values mentioned for the various physical parameters, dimensions or quantities are only approximations and it is envisaged that the values higher/lower than the numerical values assigned to the parameters, dimensions or quantities fall within the scope of the disclosure, unless there is a statement in the specification specific to the contrary.

While considerable emphasis has been placed herein on the components and component parts of the preferred embodiments, it will be appreciated that many embodiments can be made and that many changes can be made in the preferred embodiments without departing from the principles of the disclosure. These and other changes in the preferred embodiment as well as other embodiments of the disclosure will be apparent to those skilled in the art from the disclosure herein, whereby it is to

be distinctly understood that the foregoing descriptive matter is to be interpreted merely as illustrative of the disclosure and not as a limitation.

#### **TECHNICAL ADVANTAGES AND ECONOMIC SIGNIFICANCE**

- The process of the present disclosure recovers used, deactivated ionic liquid, which can be further reused for different applications.
- The process of the present disclosure saves undue waste of valuable chemicals; thereby reducing the environmental hazards.
- The expenditure on purchasing fresh ionic liquid catalyst for every single application is reduced.
- The anionic component complex obtained as a result of the process of the present disclosure may find further applications in organic light-emitting diodes.

**CLAIMS:**

1. A process for separating the cationic and anionic components from deactivated ionic liquids; said process comprising the following steps:
  - a. admixing deactivated ionic liquid in a first solvent and heating at a temperature ranging from 60 to 80 °C to obtain a first admixture;
  - b. admixing 8-hydroxyquinoline in a second solvent and heating at a temperature ranging from 60 to 80 °C to obtain a second admixture;
  - c. preparing a dispersion by admixing said first admixture with said second admixture, in a drop-wise manner, while maintaining the pH from 4 to 7 by using at least one pH adjusting agent;
  - d. cooling said dispersion to a temperature below 60 °C followed by filtering to obtain:
    - i. a residue comprising a precipitate of said anionic component and 8-hydroxyquinoline; and
    - ii. a filtrate comprising said cationic component, deactivating components of said ionic liquid and said first and second solvent;
  - e. washing said residue with said second solvent followed by drying to obtain purified precipitate;
  - f. distilling said filtrate to remove said first and second solvent and obtain a mass comprising said cationic component and said deactivating components;
  - g. extracting deactivating components from said mass by at least one extracting agent to obtain the cationic component devoid of deactivating components; and
  - h. crystallizing said cationic component by at least one crystallizing agent to recover said cationic component.
2. The process as claimed in claim 1, wherein said cationic component is selected from the group consisting of 1-butyl-3-methyl imidazolium bromide, 1-butyl-3-methyl imidazolium chloride, 1-butyl-4-methylpyridinium chloride and 1-butyl-4-methylpyridinium bromide.

3. The process as claimed in claim 1, wherein said anionic component is a metal chloride.
4. The process as claimed in claim 3, wherein said anionic component is aluminum chloride.
5. The process as claimed in claim 1, wherein the deactivating component is at least one from the group consisting of polymers, tar, hydrocarbons and moisture.
6. The process as claimed in claim 1, wherein said first solvent is toluene.
7. The process as claimed in claim 1, wherein said second solvent is ethanol.
8. The process as claimed in claim 1, wherein the step of preparing the dispersion is carried out by incorporating said first admixture into said second admixture.
9. The process as claimed in claim 1, wherein the step of preparing the dispersion is carried out by incorporating said second admixture into said first admixture.
10. The process as claimed in claim 1, wherein said pH adjusting agent is at least one from the group consisting of sodium acetate, sodium hydroxide and sodium carbonate.
11. The process as claimed in claim 1, wherein said precipitate is a tris(8-hydroquinalato) aluminum (III) complex.
12. The process as claimed in claim 1, wherein said extracting agent is at least one from the group consisting of ethylene acetate and toluene.

13. The process as claimed in claim 1, wherein said crystallizing agent is at least one from the group consisting of acetonitrile and dichloromethane.
14. The process as claimed in claim 1, wherein said precipitate is characterized as a fluorescent material for use in organic light-emitting diodes.