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(54) **Title:** A PROCESS FOR THE PURIFICATION OF CRUDE CARBOXYLIC ACID

(57) **Abstract:** The present disclosure provides a process for the purification of crude carboxylic acid. The process comprises formation of an adduct of carboxylic acid with a Lewis base. The obtained adduct is treated with at least one fluid medium to break the adduct of carboxylic acid and Lewis base to obtain purified carboxylic acid.



A PROCESS FOR THE PURIFICATION OF CRUDE CARBOXYLIC ACID

Field

The present disclosure relates to a process for the purification of crude carboxylic acid.

Background

Terephthalic acid, isophthalic acid and orthophthalic acid are the aromatic dicarboxylic acids having formula $C_6H_4(COOH)_2$. Terephthalic acid, isophthalic acid and orthophthalic acid are isomers. These colorless solid are commodity chemical and is used as a precursor for the preparation of polyester, polyethylene phthalate (PET) and the like for manufacturing clothing and high-performance multi-purpose plastics.

Commercially, terephthalic acid is produced by the oxidation of p-xylene using air or oxygen rich air as an oxidant in the presence of a catalyst and a promoter employing acetic acid as a solvent. A terephthalic acid plant comprises two major sections i.e. the oxidation and the hydrogenation section.

During the oxidation process of para-xylene, crude terephthalic acid is the main product and intermediates such as para-tolualdehyde, para-toluic acid, 4-carboxybenzaldehyde (4-CBA) and side products such as isophthalic acid, phthalic acid, meta or ortho-tolualdehyde, meta or ortho-toluic acid, 2 or 3-carboxybenzaldehyde, 3 or 4-bromo methyl benzoic acid, benzoic acid, trimellitic acid, trimesic acid, benzaldehyde, phthalaldehyde, ethylbenzaldehyde, methylstyrene, diphenic acid, 2- biphenyl carboxylic acid, hemi mellitic acid, dimethyl terephthalate, methyl p-toluate, 3-hydroxy 4-methyl benzoic acid, terephthaldehyde, styrene, phenol, toluene, benzene, ethylbenzene, methylethylbenzene, formaldehyde, 1,3-cyclopentadiene, indene, methylnaphthalene, anthracene, phenanthrene, phenylacetylene, methylbiphenyl, diphenylbutane, naphthalene, and 4,4-dimethylbibenzyl, vinylacetylene are produced. The intermediates are formed in large

quantities and eventually are converted into crude terephthalic acid during the wet oxidation of para-xylene. However, the side products formed are in small quantities.

In order to use terephthalic acid as a starting material, for example, in the preparation of polyethylene terephthalate, the content of 4-carboxybenzaldehyde (4-CBA) is recommended to be preferably below 100 ppm.

During the production of terephthalic acid, the typical range of 4-CBA produced is between 2000 ppm to 10000 ppm and para-toluic acid is produced in the range of 150 ppm to 5000 ppm. Therefore, reducing the 4-CBA impurity is very important for further use of terephthalic acid. 4-CBA, if it exists in large quantities in terephthalic acid, acts as a chain terminator during the PET polymerization process, and hence the desired PET molecular weight may not be achieved. Conventionally, crude terephthalic acid is subjected to hydrogenation to convert 4-CBA into p-toluic acid and subsequently p-toluic acid is separated.

Various techniques have been explored to purify crude terephthalic acid. Attempts have also been made to purify side products such as isophthalic acid, orthophthalic acid and the like. Conventionally used methods for the purification of crude terephthalic acid uses hydrogenation catalyst like 5% Pd/C which are costly. Further, some of the processes employ conditions that are severe and therefore, energy inefficient.

Therefore, there is felt need for simple and economic process for purification of crude carboxylic acid.

Objects

Some of the objects of the present disclosure, which at least one embodiment herein satisfies, are as follows.

It is an object of the present disclosure to ameliorate one or more problems of the prior art or to at least provide a useful alternative.

Another object of the present disclosure is to provide a process for the purification of crude carboxylic acid.

Still another object of the present disclosure is to provide a process for the purification of crude carboxylic acid to produce purified carboxylic acid having low 4-CBA content.

Yet another object of the present disclosure is to provide a process to separate an adduct of Lewis based-carboxylic acid selectively.

Still another object of the present disclosure is to provide an environment friendly, simple, safe and cost effective process for purification of carboxylic acid.

Another object of the present disclosure to provide a process for the purification of crude carboxylic acid by reducing the metallic and other impurities to less than or equal to the standard specification; thereby increasing the industrial applicability of the resulting purified acid.

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 the purification of crude carboxylic acid, the process comprising the step of reacting at least one Lewis base with at least one crude carboxylic acid at a temperature ranging from 50 °C to 200 °C and at a pressure ranging from 1 to 10 bar to obtain a clear solution (resultant mass) comprising Lewis base-carboxylic acid adducts in dissolved form. The so obtained solution (resultant mass) undergoes crystallization to obtain at least one adduct of carboxylic acid at a temperature lower than the temperature at which the reaction of Lewis base with crude

carboxylic acids is carried out. Lewis base-carboxylic acid adduct is at least one selected from the group consisting of Lewis base-terephthalic acid. The crystals obtained after the step of crystallization are selectively separated from the resultant mass to obtain crystals of the Lewis base-carboxylic acid adduct. The separated adduct is then treated with at least one fluid medium to obtain purified carboxylic acid.

Brief Description of the Accompanying Drawing

Figure 1 is an exemplary illustration of the process for producing purified carboxylic acid from crude carboxylic acid in accordance with the present disclosure.

Detailed Description

In one aspect, the present disclosure provides a process for the purification of crude carboxylic acid. The process is described below.

Crude carboxylic acid is reacted with at least one Lewis base by heating in the temperature range of 50 °C to 200 °C and at a pressure ranging from 1 to 10 bar to obtain adducts of Lewis base and carboxylic acid. The obtained Lewis base-carboxylic acid adduct is selected from the group consisting of Lewis base-terephthalic acid adduct in a dissolved form in a resultant mass. The so obtained adduct appears as homogenous solution, i.e. it is completely dissolved in a resultant mass.

In the present disclosure, the crude carboxylic acid is a crude terephthalic acid.

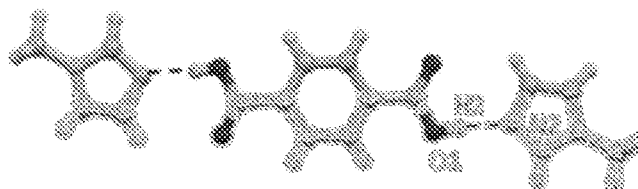
Lewis base that can be used in the present disclosure include substituted and non-substituted linear, branched, cyclic, polycyclic, heterocyclic and heteroaryl Lewis base and any combination thereof. Non-limiting examples of the Lewis base include linear and branched substituted and non-substituted mono, di and trialkyl amine and phosphine, linear and branched substituted and non-substituted mono and dialkyl sulfide, substituted and non-substituted imidazole, pyrazole, thiazole, isothiazole, azathiazole, oxothiazole, oxazine, oxazoline, oxazaborole, dithiazole, triazole, selenozole, oxaphosphole, pyrrole, borole, furan, thiophene, phosphole, pentazole, indole, indoline, oxazole, isothiazole, tetrazole, benzofuran, dibenzofuran,

benzothiophene, dibenzothiophene, thiadiazole, pyridine, pyrimidine, pyrazine, pyridazine, piperazine, piperidine, morpholine, pyran, aniline, phthalazine, quinazoline, quinoxaline, tromethamine, triethanoamine, pyrrolidine 1-(2-hydroxyethyl), morpholine 4-(2-hydroxyethyl), L-Lysine, hydrabamine, N-methyl glucamine, ethylene diamine, ethanoamine, 2-dimethylamino ethanol, diethanolamine, deanol, choline, benzathine, benethamine, L-arginine, ammonia and any combination thereof.

In an exemplary embodiment the Lewis base is 1-methyl imidazole. In one embodiment of the present disclosure, 1-methyl imidazole is reacted with crude terephthalic acid to form 1-methyl imidazole terephthalic acid adduct.

The adduct formation takes place due to dipole-dipole interaction between the nitrogen atom of the 1-methyl imidazole and hydrogen atom of the carboxylic acids group present in terephthalic acid.

In an embodiment dipole-dipole interaction between the nitrogen atom of the 1-methyl imidazole and hydrogen atom of terephthalic acid is represented below in Formula-I:



Formula-I

In another embodiment of the present disclosure, the mole ratio of Lewis base to crude carboxylic acid varies from 1:1 to 50:1, more particularly from 2:1 to 12:1 and even more particularly from 6:1 to 10:1. The quantity of crude carboxylic acid to Lewis base ratio is such that all adducts formed of carboxylic acid, its intermediates and impurities remain soluble in the resultant mass at a predetermined temperature.

The so obtained homogenous (clear) solution of the resultant mass containing the adducts of Lewis base-carboxylic acid undergoes crystallization at a temperature lower than the temperature at which the reaction of Lewis base with a crude

carboxylic acid (adduct formation) is carried out to obtain the crystals of at least one adduct of the Lewis base-carboxylic acid. In an exemplary embodiment, the adduct of Lewis base-carboxylic acid is the adduct of 1-methyl imidazole- terephthalic acid.

The crystals obtained in above step are separated by filtering the solids from the mother liquor or decanting the mother liquor followed by washing the crystals optionally with the fluid medium to obtain separated crystals of Lewis base-carboxylic acid. In an exemplary embodiment, the separated crystals are the crystals of 1-methyl imidazole- terephthalic acid adduct.

The separated crystals of Lewis base-carboxylic acid are treated with at least one first fluid medium to obtain purified carboxylic acid. The separation (de-adduction) is carried out by stirring the crystals of the adduct in the first fluid medium, either at room temperature or optionally heating the mixture of the first fluid medium and the crystals of Lewis base-carboxylic acid adduct at a temperature ranging from 50 °C to 140 °C.

The first fluid medium is at least one selected from the group comprising methanol, ethanol, propanol, benzyl alcohol, water and combinations thereof. The first fluid medium can be recovered after the step of de-adduction.

In the present disclosure, the washing fluid medium is referred as the second fluid medium. The second fluid medium is selected from the group consisting of Lewis bases, aliphatic and aromatic solvents including alkane solvents, aromatic hydrocarbon solvents, ethers, fluorinated and chlorinated hydrocarbons, esters, ethers, ketones, and acids. In an embodiment, the second fluid medium is at least one selected from the group consisting of methyl acetate, ethyl acetate, acetonitrile, dichloromethane, dichloroethane, 1-methyl imidazole and combination thereof. The separated adduct was washed with the second fluid medium to remove/reduce the different impurities adhered to surface of the adduct of terephthalic acid -lewis base.

The fluid media and chemicals used in the process of the present disclosure can be recovered for further use.

In the present disclosure, the process for preparing adduct is carried out in a reactor. Non-limiting examples of reactors for the formation of the complex or adduct include single or series of continuous stirred flow reactors, static mixers, plug flow reactors, fixed bed reactors, fluidized bed reactors, packed bed reactors and any combination thereof.

In another aspect of the present disclosure, the process is described hereinafter, as depicted in Figure 1. Predetermined quantity of crude terephthalic acid and Lewis base is fed to the reaction section via line **1** and **2** respectively. Crude terephthalic acid is charged using a solid conveying system.

The adduct undergoes crystallization to obtain at least one adduct of carboxylic acid selected from the group consisting of Lewis base terephthalic acid adduct at a temperature lower than the temperature at which the reaction of Lewis base with crude carboxylic acid is carried out. A homogeneous solution of the adduct of carboxylic acid (**A**) dissolved in a Lewis base is sent to crystallization section (**B**) via line **3** to selectively crystallizes of the adduct of carboxylic acid and Lewis base.

Crystallization section comprises a single crystallizer or series of crystallizers and non-limiting examples of crystallizers include Forced Circulation crystallizers, Draft Tube Baffle crystallizers, Oslo Crystallizers, and the like.

The crystals obtained after the step of crystallization are selectively separated from the resultant mass to obtain crystals of adduct of Lewis base carboxylic acid.

A slurry stream from crystallization section is transformed to the filtration section (**C**) via line **4** to separate the adduct of carboxylic acid and Lewis base from the excess Lewis base.

The filtration section comprises a single filter or series of filters and non-limiting examples of the filtration system include all types of filters like belt filters, rotary filters, Nutsche filters, and the like, operated under positive, negative or atmospheric pressure.

The filtrate can be divided into two parts, one part is recycled back to the reaction section (A) via line 17 and the other part goes to reactant recovery section (G) via line 16.

The adduct is washed with the second fluid medium to obtain crystals of Lewis-base-carboxylic acid adduct. The adduct is transformed to a washing section (D) via line 5 where the adduct is washed using second fluid medium (M) coming from line no 6.

The washing section comprises a reactor and a filtration system and other necessary unit operations and equipment(s). The washed filtrate (D) is divided into two parts, one part is recycled back via line 14 to the washing section (D) and the other part goes to the separation of second fluid medium and Lewis base and others via line 13 to the solvent recovery section (H).

The second fluid medium used for washing the adduct in the present disclosure includes molecular solvents having 1 to 12 carbon atoms. The second fluid medium is selected from the group consisting of Lewis bases, aliphatic or aromatic solvents like alkane solvents, aromatic hydrocarbon solvents, ethers, fluorinated and chlorinated hydrocarbons, esters, ethers, ketones, alcohols, acids, water and any combination thereof.

In an exemplary embodiment, examples of the second fluid medium includes but are not limited to, methyl acetate, ethyl acetate, dichloromethane, dichloroethane, acetonitrile, 1-methyl imidazole and any combination thereof.

The ratio of second fluid medium to adduct varies from 1:1 to 50:1, preferably from 1:1 to 8:1.

In one embodiment of the present disclosure, the adduct washing is carried out in a temperature range of 50 °C to 200 °C and 1 to 10 bar pressure.

In another embodiment of the present disclosure, the Lewis base used for the adduct formation and the second fluid medium for washing the adduct are the same.

In yet another embodiment of the present disclosure, the Lewis base used for the adduct formation and the second fluid medium used for washing the adduct is 1-methyl imidazole.

The washed adduct (**D**) via line **7** is fed to the de-adduct section (**E**) for terephthalic regeneration where the first fluid medium (**N**) is charged via line **8**.

After washing, the adduct is treated with a fluid medium capable of breaking the adduct of Lewis base and terephthalic acid, which is termed as a first fluid medium in the present disclosure.

Non limiting examples of the first fluid medium include aliphatic and aromatic alcohol, water, and other polar solvents.

Exemplary examples of the first fluid medium include methanol, ethanol, propanol, benzyl alcohol, water and any combination thereof.

In one embodiment of the present disclosure, the regeneration or reconstitution or adduct breaking is carried out at a temperature range of 50 °C to 140 °C and 1 to 10 bar pressure.

De-adduct section comprises one crystallizer or a series of crystallizers or can be a combination of crystallizer followed by reactor for washing the purified terephthalic acid using the first fluid medium or other solvents like water and methyl acetate. After de-adduction, the first fluid medium is recycled back via line **11** to the solvent recovery section (**I**) and the recovered solvent can be used for further de-adduction sent via line **12**.

The wet purified carboxylic acid is transformed via line **9** to the drying section (**F**) to obtain dry purified carboxylic acid (**L**) of desired quality and specifications and is recovered from line **10**.

The drying section (**F**) comprises one or a series of reactors and non-limiting examples of dryers include fluidized bed, atmospheric tray, vacuum tray, agitated bed, direct rotary, indirect rotary, spray, spouted bed, vibrated bed, drum, belt, plate, vacuum, disc, paddle, column, filter, ring, jet-zone, microwave, freeze, solar and any

combination thereof. Relative motion between the drying medium and the solid to be dried can be concurrent, counter current and mixed flow.

The Lewis base is recycled back via line **17** and **18** to the reaction section. A Lewis base coming from the solvent recovery section (**H**), the solvent recovery section (**I**) and the intermediate recovery section (**J**) goes to the reactant recovery section via line **19**, **20** and **22** respectively.

Adduct of intermediates with Lewis base is charged to intermediate recovery section (**J**) via line **21**. Recovered intermediates (**K**) are recycled back to the oxidation reaction section via line **23**.

In one embodiment of the present disclosure, the recovered Lewis base, first fluid medium and second fluid medium are recycled and reused.

In another embodiment of the present disclosure, a process has been developed to remove undesired impurities from the desired carboxylic acid such as carboxy benzaldehyde, toluic acid and the like and the removal of metal catalyst in benzene dicarboxylic acid production.

In another embodiment of the present disclosure, the adduct formation is carried out in single or multiple stages to get the desired purity and specification of terephthalic acid.

In still another embodiment of the present disclosure, the adduct formed is re-dissolved in fresh or recycled Lewis base in single or multiple stages to achieve the desired purity and specification of terephthalic acid.

After dissolution, the adduct is separated from the excess Lewis base by cooling or anti-solvent crystallization and combination thereof.

The present disclosure is further illustrated herein below with the help of the following experiments. The experiments 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 embodiments herein. The present disclosure is further described in light of the following experiments 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.

Experimental details:

In this experiment, the reaction between crude carboxylic acid and 1-methyl imidazole was carried out to prepare an adduct of carboxylic acid with Lewis base and its selective separation. The washing fluid medium used was dichloromethane and the de-adduction medium (First fluid medium) used was methanol. The setup consisted of a round bottom flask into which crude carboxylic acid and 1-methyl imidazole were added. Optionally acetonitrile was also added to the flask and reaction was carried out under reflux with stirring. After completion of the reaction, the reaction mixture was cooled and solid adduct was obtained. The solid adduct was filtered and washed with dichloromethane to remove unreacted 1-methyl imidazole. A white coloured adduct was obtained. Methanol was used as the first fluid medium to break the adduct of carboxylic acid and 1-methyl imidazole to regenerate purified carboxylic acid.

The 4-CBA content was reduced in the purified carboxylic acid as compared to the crude carboxylic acid.

Example 1: Process for the purification of terephthalic acid in accordance with the present disclosure

49.8 grams of terephthalic acid was mixed with 147.78 grams of 1-methyl imidazole in 1:6 mole ratio at 105 °C. A homogenous/clear solution was obtained. The obtained homogenous solution was cooled to 27-30 °C, solid adduct was obtained (crystalized out) which was filtered out and washed with methyl acetate. The solid of adduct was white in color. The obtained adduct was treated with methanol as a solvent to break the adduct of terephthalic acid and 1-methyl imidazole at 27 °C to obtain pure terephthalic acid.

The 4-CBA content in the purified terephthalic acid was reduced to 143 ppm from its initial content in crude terephthalic acid of 2900 ppm.

Example 2: Comparative example

49.8 grams of terephthalic acid was mixed with 178.44 grams of N-methyl pyrrolidone in 1:6 mole ratio at temperature 105 °C. No clear solution was observed at this temperature. Hence, the temperature was further raised to get a clear solution which was obtained at 140 °C. At this temperature, N-methyl pyrrolidone colour turned to dark brown from colourless liquid.

The liquid so obtained was kept cooled to 27-30 °C. The solid adduct was obtained which was filtered out and washed with N-methyl pyrrolidone to remove/reduce the colour of adduct which was brown in colour after reaction.

Pure terephthalic acid was regenerated from the adduct using methanol as solvent . The adduct upon treating with methanol broke into pure terephthalic acid and N-methyl pyrrolidone at 27 °C.

The 4-CBA content reduced to 1200 ppm from its initial content in crude terephthalic acid of 2900 ppm.

Example 3: Process for reducing the undesired impurities of the metal catalyst and purification of terephthalic acid in accordance with the present disclosure

Example 3 was carried out similar to example 2 except adduct formed in first stage of example 2 with N-methyl pyrrolidone was again dissolved in 147.78 gms 1-methyl imidazole at 105 °C to obtain homogenous solution. The homogenous solution was cooled to 27-30 °C, to obtain adduct crystals. The obtained crystals were filtered and washed with methyl acetate to get white colour adduct.

Pure terephthalic acid was regenerated from the above adduct using methanol as solvent to break the adduct of terephthalic acid and 1-methyl imidazole at 27 °C.

The 4-CBA content reduced to 10 ppm from its initial content in crude terephthalic acid of 2900 ppm. Also, the metal content in pure terephthalic acid particularly Co, Mn & Ni was reduced to less than or equal to 1 ppm from its initial content of around 25 ppm in crude terephthalic acid respectively.

Technical advances

The process of the present disclosure described herein above has several technical advantages including, but not limited to, the realization of:

- generating purified terephthalic acid with the required specification of 4-CBA.
- purification of crude terephthalic acid using moderate temperature and atmospheric pressure.
- purification of crude terephthalic acid using reusable and recyclable chemicals.

The exemplary embodiments herein quantifies the benefits arising out of this disclosure and the various features and advantageous details thereof are explained with reference to the non-limiting embodiments in the 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 foregoing description of the specific embodiments reveals 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 has 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.

Any discussion of documents, acts, materials, devices, articles and 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.

While considerable emphasis has been placed herein on the particular features of this disclosure, it will be appreciated that various modifications 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 modifications in the nature of the disclosure or the preferred embodiments 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.

CLAIMS:

1. A process for the purification of crude carboxylic acid, said process comprising the following steps:
 - a. reacting at least one Lewis base with at least one crude carboxylic acid at a temperature ranging from 50 °C to 200 °C and at a pressure ranging from 1 to 10 bar to obtain Lewis base-carboxylic acid adduct in a dissolved form in a resultant mass;
 - b. crystallizing at least one Lewis base-carboxylic acid adduct at a temperature lower than the temperature at which the reaction in step (a) is carried out to obtain crystals of the Lewis base-carboxylic acid adduct;
 - c. separating said crystals of the Lewis base-carboxylic acid adduct from the resultant mass to obtain separated crystals of the Lewis base-carboxylic acid adduct; and
 - d. treating said separated crystals of the Lewis base-carboxylic acid adduct with at least one first fluid medium to obtain purified carboxylic acid.
2. The process as claimed in claim 1, wherein the crude carboxylic acid is crude terephthalic acid.
3. The process as claimed in claim 1, wherein the Lewis base is at least one selected from the group comprising substituted and non-substituted linear, branched, cyclic, polycyclic, heterocyclic and heteroaromatic Lewis base.
4. The process as claimed in claim 1, wherein the Lewis base is 1-methyl imidazole.
5. The process as claimed in claim 1, wherein the first fluid medium is at least one selected from the group comprising methanol, ethanol, propanol, benzyl alcohol, water and combination thereof.
6. The process as claimed in claim 1, wherein the mole ratio of said Lewis base to said crude carboxylic acid ranges from 1:1 to 50:1.
7. The process as claimed in claim 1, wherein said at least one carboxylic acid adduct of step (b) is Lewis base-terephthalic acid adduct.

8. The process as claimed in claim 1, wherein said crystals of the adduct of carboxylic acids and Lewis base are subjected to washing with second fluid medium prior to breaking the adduct of carboxylic acid and Lewis base.
9. The process as claimed in claim 8, wherein the second fluid medium is at least one selected from the group comprising methyl acetate, ethyl acetate, acetonitrile, dichloromethane, dichloroethane, 1-methyl imidazole and combinations thereof.
10. The process as claimed in claim 1, wherein said step of treating said adduct with said first fluid medium further comprises heating the mixture of said first fluid medium and said crystals of the Lewis base-carboxylic acid adduct at a temperature ranging from 20 °C to 250 °C to obtain purified carboxylic acid.
11. The process as claimed in claim 1, wherein the Lewis base remaining after the step of separation of said crystals of the Lewis base-carboxylic acid adduct is recovered and recycled.
12. A terephthalic acid having 4-CBA content less than 200 ppm prepared by the process as claimed in claim 1.

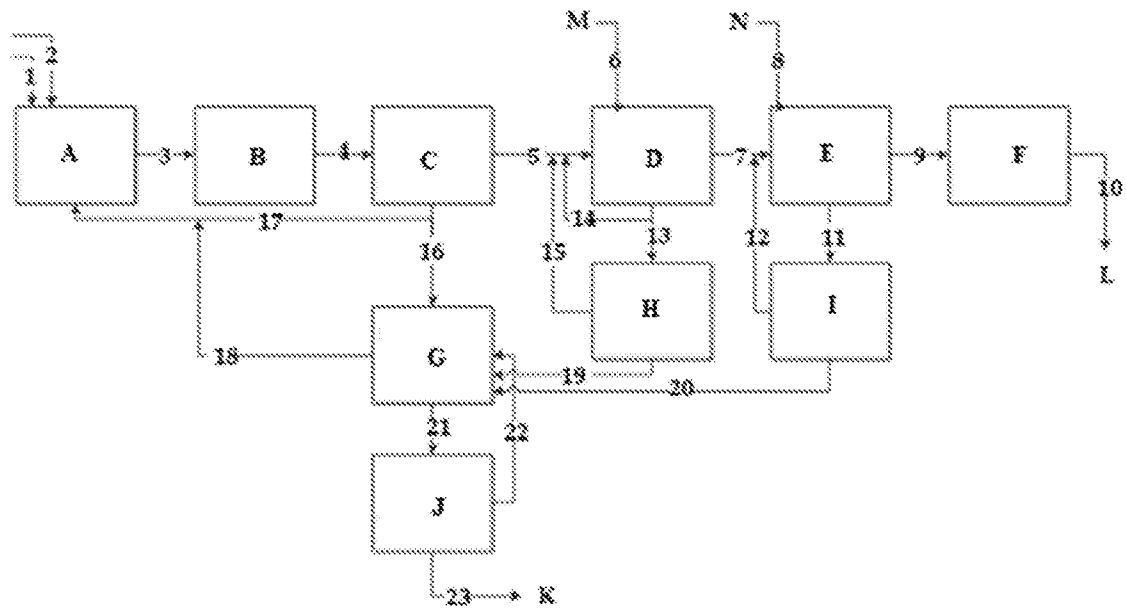


FIGURE 1

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2015/054190

A. CLASSIFICATION OF SUBJECT MATTER
C07C55/26, C07C55/02, C07C51/34 Version=2015.01

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C07C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

IPO INTERNAL DATABASE, PATSEER

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US20120004456 A1 (UOP LLC) & 05 Jan 2012 whole document	1-12
A	WO2014058943 A1 (UOP LLC) & 17 April 2014 whole document	1-12

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A" document defining the general state of the art which is not considered to be of particular relevance	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
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