



(51) International Patent Classification:

C07C 7/00 (2006.01) **B01J 20/00** (2006.01)
B01D 15/00 (2006.01)

(21) International Application Number:

PCT/IB2015/057118

(22) International Filing Date:

22 September 2015 (22.09.2015)

(25) Filing Language:

English

(26) Publication Language:

English

(30) Priority Data:

2959/MUM/2014 16 September 2014 (16.09.2014) IN

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400001 (IN).(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:

- with international search report (Art. 21(3))
- before the expiration of the time limit for amending the
claims and to be republished in the event of receipt of
amendments (Rule 48.2(h))
- with information concerning request for restoration of the
right of priority in respect of one or more priority claims;
the decision of the receiving Office regarding the request
for restoration is pending and will be published separately
once available (Rules 26bis.3 and 48.2(j))

(54) Title: CONTINUOUS PROCESS FOR REMOVING IMPURITIES FROM IMPURE HEAVY ALKYLATED BENZENE (HAB) AND REGENERATING AN EXHAUSTED ADSORBENT

(57) Abstract: The present disclosure provides a process for the removal of impurities from impure heavy alkylated benzene (HAB) using an adsorbent. The adsorbent can then be regenerated by using a non-carcinogenic and eco-friendly fluid medium, which can be recovered and reused. The process of the present disclosure avoids the use of steam for regeneration/displacement of impurities from the adsorbent and hence, minimizes effluent generation having higher COD.



WO 2016/046695 A1

CONTINUOUS PROCESS FOR REMOVING IMPURITIES FROM IMPURE HEAVY ALKYLATED BENZENE (HAB) AND REGENERATING AN EXHAUSTED ADSORBENT

FIELD

- 5 The present disclosure relates to a process for removing impurities and regenerating an adsorbent.

DEFINITIONS

- As used in the present disclosure, the following words and phrases are generally intended to have the meaning as set forth below, except to the extent that the context in
10 which they are used indicate otherwise.

The expression 'HAB' for the purpose of the present disclosure refers to heavy alkyl benzene or heavy alkylated benzene.

- The expression 'ASTM No' for the purpose of the present disclosure refers to the intensity of color, as tested by the American Society for Testing and Materials
15 (ASTM), particularly for petrochemical and refining industry.

The expression 'Saybolt No.' for the purpose of the present disclosure refers to a color testing method for the products with intensity less than 0.5 ASTM (ASTM D-156).

BACKGROUND

- Heavy alkylated benzene (HAB) is produced during alkylation of benzene with
20 olefin(s) and primarily contains dialkyl benzenes and di-phenyl alkanes with small quantities of condensed ring aromatic like methyl anthracenes, tetralins and indanes. The formation of dialkyl benzenes can be suppressed up to some extent by taking a high ratio of benzene to olefin. Di/tri-phenyl alkanes are generated due to the presence of di, tri-olefins -during the alkylation reaction. The other compounds may either come
25 from linear alkyl benzene (LAB) feed stock, formed during the alkylation reaction or separation process.

HAB, which is a by-product in LAB plants, has a yellowish color. The C=C bonds in conjugation with aromatic rings are chromophoric in nature. In HAB, poly aromatic rings with C=C double bond in the alkyl side chain may also conjugate with the π -electron cloud of the aromatic ring and could be one of the reasons for the coloring in
5 HAB.

Usually, sulfuric acid treatment and adsorption techniques are used to remove the color causing compounds from HAB. However, sulfuric acid treatment method is hazardous and the post treatment process involves the use of excess acid followed by washing with water to remove the acid and finally drying using nitrogen. This results in acid
10 sludge formation which needs to be disposed.

Accordingly, there is a need for a simple, cost efficient, and environment friendly process for removing impurities from impure heavy alkylated benzene and regenerating the adsorbent.

OBJECTS

15 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.

An object of the present disclosure is to provide a simple and efficient process for
20 removing impurities from heavy alkylated benzene.

Another object of the present disclosure is to provide a continuous process for removing impurities and regenerating the adsorbent.

Still another object of the present disclosure is to provide a process for regenerating an exhausted adsorbent by using an eco-friendly and non-carcinogenic fluid medium.

25 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 removal of impurities from impure heavy alkylated benzene by using an adsorbent and regeneration of an exhausted adsorbent. In the process of the present disclosure impure heavy alkylated benzene is passed through an adsorbent to obtain heavy alkylated benzene substantially free from impurities. The impurities from the heavy alkylated benzene get adsorbed on the adsorbent. Typically the adsorbent is activated carbon. To regenerate the exhausted adsorbent, at least one fluid medium is passed through the exhausted adsorbent for displacing the impurities adsorbed on the adsorbent. In this step, the adsorbent may contain some residual fluid medium adsorbed on it.

Further, for removing the residual fluid medium adsorbed on the adsorbent, at least one inert gas is passed through the adsorbent at a temperature in the range of 180 °C to 220 °C for 4 hours to 6 hours to obtain regenerated adsorbent.

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 following 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 has been described in terms of preferred
5 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.

Linear alkyl benzene (LAB) plant produces linear alkyl benzene via alkylation of benzene with olefins of 10 to 14 carbon atoms by using hydrofluoric acid. In this
10 process, around 10 to 12 % heavy alkyl benzene (HAB) is also produced along with detergent grade linear alkyl benzene. HAB is separated from detergent-quality linear alkyl benzene by conventional separation methods such as distillation. The HAB obtained as a by-product of LAB plant is impure as it has a yellowish color due to the presence of color causing compounds. Conventionally, sulfuric acid treatment and
15 adsorption techniques are used to remove the color causing compounds from the impure HAB.

Generally, LAB plants carry out re-processing of impure HAB (colored HAB) by mixing it with the feed to an alkylation reactor. This material then moves through all the distillation columns of the alkylation unit along with the normal feed to alkylation,
20 resulting in additional operating cost. In order to ensure that impure HAB does not disturb the normal operating conditions, the feed rate is kept extremely low as compared to the normal processing rate. The lowering of the feed to alkylation reduces the production of the LAB plant.

Conventionally, the adsorptive process used, employ clay and carbon as adsorbents.
25 These adsorbents are normally used only once and released in the landfills after they get saturated with impurities. This results in environmental pollution. In some cases, regeneration of the saturated adsorbents is also carried out by using steam or solvent such as benzene. The regeneration of adsorbent using carcinogenic solvent (benzene) is not advisable and the regeneration using steam produces significant amount of waste
30 water having high COD (Chemical Oxygen Demand) which is fatal for aquatic life.

Accordingly, the present disclosure provides a continuous process for removing color causing compounds from impure heavy alkylated benzene (HAB) and regenerating the exhausted adsorbent in a simple and efficient way.

5 In the first step, a stream of impure HAB is passed through a packed bed reactor having an adsorbent. The impure heavy alkylated benzene can be passed through said adsorbent at a flow rate ranging from 1.4 ml/min to 2 ml/min. The adsorbent is used to remove the impurities from the impure HAB. Impure HAB is the by-product obtained in linear alkyl benzene (LAB) plant and has a yellowish color. The process is carried out at ambient temperature in a continuous operation.

10 In one embodiment of the present disclosure activated carbon is used as the adsorbent to remove the impurities from impure HAB. The impurities in HAB mainly comprise color causing compounds such as oxygenates, conjugated dienes, polyaromatic rings with C=C double bond in the alkyl side chain which may conjugate with the π electron cloud of the aromatic ring and the like.

15 In one embodiment the color causing compounds are adsorbed on the adsorbent and the heavy alkylated benzene so obtained is substantially free from impurities. The color causing compounds adsorbed on the adsorbent limit the activity of the adsorbent to adsorb further impurities and hence it is referred to as exhausted adsorbent.

In the second step, the adsorbent is regenerated. The impurities of the exhausted
20 adsorbent are displaced from the exhausted adsorbent by passing a stream of at least one fluid medium through the exhausted adsorbent. Non-limiting examples of fluid media that can be used to displace the color causing compounds from exhausted adsorbent include heavy normal paraffin (HNP) and n-nonane. The amount of said fluid medium can be in the range of 320 gm to 350 gm, with respect to the adsorbent. The
25 stream of fluid medium can be passed through said exhausted adsorbent at a flow rate ranging from 1 ml/min to 2.1 ml/min.

Displacement of the adsorbed color impurities is carried out at ambient temperature. In one embodiment the color impurities are solubilized in the fluid medium and hence displace the color impurities from the adsorbent.

The fluid medium used for displacing the color causing compounds from the adsorbent is continuously passed through the adsorbent bed till the Saybolt No. /ASTM No. of the inlet and outlet of the fluid medium becomes the same. Initially, there is a decrease in the Saybolt No. /ASTM No. as the color causing compounds are washed off in the fluid medium. Eventually, all the color causing compounds are washed off and the Saybolt No. /ASTM No. of the fluid medium increases; and the Saybolt No. /ASTM No. of the inlet and outlet of the fluid medium become the same.

The impure heavy alkylated benzene before treating with adsorbent can have Saybolt No/ASTM color in the range of (-) 1 to (-) 16. Further, after the adsorption process the heavy alkylated benzene substantially free from impurities has Saybolt No/ASTM color in the range of (+) 16 to (+) 25.

In an embodiment of the present disclosure, the impurities present in the fluid medium are separated by distillation and the impurity free fluid medium is recovered and reused in the next cycle.

In the third step, after the impurities have been displaced/ removed from the adsorbent, some residual amount of fluid medium still remains in the intra and inter particle voids/pores of the adsorbent. In an embodiment of the present disclosure, an inert gas such as nitrogen or argon is passed through the adsorbent at a temperature ranging from 180 °C to 220 °C for 4 to 6 hours to remove the molecules of the fluid medium from the adsorbent, thus, regenerating the adsorbent which can be used in the next cycle.

In one embodiment, the regenerated adsorbent has 80-90% activity as compared to the adsorbent which was used originally (fresh adsorbent).

Regeneration of the adsorbent bed is confirmed by carrying out the Saybolt and/or ASTM color analysis of the HNP collected at regular interval of time.

The removal of the impurities from impure HAB by using the process of the present disclosure can be carried out continuously at ambient temperature. The process uses non-carcinogenic, eco-friendly fluid media like HNP and n-nonane; and the fluid medium can be recovered and reused. An improvement from (-) 16 to (+) 16 in the

Saybolt - color analysis can be achieved by using the process in accordance with the present disclosure.

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 DEATAILS:

Experiment 1: Removal of color impurities from heavy alkylated benzene and regeneration of activated carbon in accordance with present disclosure:

15 A. Removal of color impurities from heavy alkylated benzene-

A stream of impure heavy alkylated benzene (HAB) having color impurities (-) 16 (measured by Saybolt No./ASTM No.) was passed through 94.79 gm of activated carbon at 30 °C, at flow rate of 1.4 ml/min. The impurities from impure HAB were adsorbed on the activated carbon (exhausted activated carbon) and a stream of heavy alkylated benzene was obtained which was substantially free from impurities having a color of (+) 16, measured by Saybolt No./ASTM No.

The results of HAB feed over activated carbon are tabulated in **Table 1:-**

Operating conditions:

- Adsorbent: Activated carbon= 94.79 gm (Particle size between 8-12 BSS/1.4 to 2.0 mm);
- Volume of reactor = 199.14 ml;
- Temperature = Ambient; and
- Flow rate = 1.4 ml/min; WHSV = 0.8 h^{-1} ; LHSV = 0.423 h^{-1} .

Table 1

HAB Sample No.	HAB Fraction quantity (mL)	Total HAB feed processed (mL)	HAB Feed processed (ml/g)	Saybolt No	ASTM
Feed				(-) 16	1.1
1	1410	1410	14.87	(+) 16	<0.5
2	360	1770	18.04	(+) 13	<0.5
3	280	2050	21.63	(+) 10	0.3
4	295	2345	24.74	(+) 13	<0.5
5	400	2745	28.96	(+) 9	0.3
6	1300	4045	42.67	(+) 10	0.3
Mixture of HAB Samples: 1 to 6				(+) 11	0.3
7	165	4210	44.41	(+) 7	0.3
8	510	4720	49.79	(+) 6	0.4
9	1300	6020	63.51	(+) 5	0.4
10	80	6100	64.35	(+) 4	0.4
Mixture of HAB Samples: 1 to 10				(+) 9	0.3
11	610	6710	70.79	(+) 4	0.4
12	1350	8060	85.03	(+) 4	0.4
Mixture of HAB Samples: 1 to 12				(+) 5	0.4
13	80	8140	85.87	(+) 3	0.4
14	350	8490	89.57	(+) 3	0.4
15	1300	9790	103.28	(+) 4	0.4
16	1300	11090	117.00	(+) 0	0.4

B. Regeneration of activated carbon-

- To regenerate the exhausted activated carbon, a stream of heavy normal paraffin (HNP) at a flow rate 2.1 ml/min was passed through the exhausted activated carbon to obtain an activated carbon substantially free from impurities, containing adsorbed HNP. The impurities from the activated carbon were washed off in heavy normal paraffin. Washing with heavy normal paraffin was carried out till the Saybolt No./ASTM No of inlet and outlet HNP became the same.
- 10 Nitrogen gas at 200 °C was passed through the activated carbon containing adsorbed HNP to obtain regenerated activated carbon. The regenerated activated carbon regained 85% of its activity.

Experiment 2: Removal of color impurities from heavy alkylated benzene and regeneration of activated carbon in accordance with present disclosure:

A similar experiment as described in experiment 1 was carried out by using n-nonane as fluid medium instead of heavy normal paraffin.

5 Experiment 3: Removal of color impurities from heavy alkylated benzene and regeneration of activated carbon in accordance with present disclosure:

A similar experiment as described in experiment 1 was carried out by using mixture of heavy normal paraffin and n-nonane as fluid medium instead of heavy normal paraffin.

The results obtained from experiment 1-3 are given in **Table 2** below.

10 Table 2:

Experiment No.	Fluid medium	Regeneration of activated carbon	Saybolt No./ASTM No (before)	Saybolt No./ASTM No (after)
1	Heavy normal paraffin (HNP)	85%	(-) 16	(+) 14
2	n-nonane	85%	(-) 16	(+)14
3	Heavy normal paraffin + n-nonane (50:50)	85%	(-) 16	(+)14

From the above experimental results it is observed that, the color impurities are removed from impure HAB and regeneration of exhausted activated carbon with regained activity of 85% is achieved.

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
5 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
10 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
15 illustrative of the disclosure and not as a limitation.

TECHNICAL ADVANCES AND ECONOMICAL SIGNIFICANCE

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

- 20 – simple and economic process for the removal of impurities from impure HAB using adsorbents;
- regeneration of adsorbent is carried out in the absence of carcinogenic solvents;
- regeneration of the adsorbent is carried out without steam and therefore, the process avoids generation of effluents having higher COD; and
- 25 – regenerated adsorbent is 85% active as compared to fresh adsorbent.

CLAIMS:

1. A continuous process for removing impurities from impure heavy alkylated benzene (HAB) using an adsorbent and regenerating an exhausted adsorbent, said process comprising:
 - 5 a. passing a stream of impure heavy alkylated benzene through the adsorbent to obtain a stream of heavy alkylated benzene substantially free from impurities and the exhausted adsorbent;
 - b. passing a stream of at least one fluid medium through said exhausted adsorbent to obtain an adsorbent substantially free from said impurities,
10 wherein said fluid medium is at least one selected from the group consisting of heavy normal paraffin (HNP) and n-nonane; and
 - c. passing at least one inert gas through said adsorbent substantially free from said impurities at a temperature in the range of 180 °C to 220 °C for 4 to 6 hours to obtain regenerated adsorbent.
- 15 2. The process as claimed in claim 1, wherein said adsorbent is activated carbon.
3. The process as claimed in claim 1, wherein said inert gas is nitrogen.
- 20 4. The process as claimed in claim 1, wherein said regenerated adsorbent has 80% to 90% of its original activity.
5. The process as claimed in claim 1, wherein said impure heavy alkylated benzene has Saybolt No/ASTM No. in the range of (-) 1 to (-) 16; and wherein
25 said heavy alkylated benzene substantially free from impurities has Saybolt No/ASTM No. in the range of (+) 16 to (+) 25.
6. The process as claimed in claim 1, wherein the amount of said fluid medium is in the range of 320 g to 350 g with respect to said adsorbent.
- 30

7. The process as claimed in claim 1, wherein said stream of impure heavy alkylated benzene is passed through said adsorbent at a flow rate ranging from 1.4 ml/min.
- 5 8. The process as claimed in claim 1, wherein said stream of at least one fluid medium is passed through said exhausted adsorbent at a flow rate ranging from 2.1 ml/min.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/IB2015/057118

A. CLASSIFICATION OF SUBJECT MATTER
C07C7/00, B01D15/00, B01J20/00 Version=2016.01

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, B01D, B01J

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)

Patseer and IPO internal database

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US20090299091A1, Petroquimica Espanola, S.A. (Petresa), Madrid (ES); Dec. 3, 2009 (03/12/2009) para 0066	7



Further documents are listed in the continuation of Box C.



See patent family annex.

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Date of the actual completion of the international search

13-01-2016

Date of mailing of the international search report

13-01-2016

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/IB2015/057118

Citation	Pub.Date	Family	Pub.Date
US 20090299091 A1	03-12-2009	EP 2006281 A4	07-07-2010
		CA 2646247 C	19-05-2015