

Description

This invention relates to a process for the removal of carbon dioxide from a gaseous stream containing at least one C_2 to C_4 olefin.

Alumina is a well known adsorbent in many chemical processes such as the polymerization of olefins, e.g. ethylene, for the removal of water and small concentrations of methanol, carbonyl-containing compounds, and peroxides.

However, the use of alumina has disadvantages that impair its effectiveness as an adsorbent. For example, alumina does not always work as an adsorbent for the removal of CO_2 , a well known catalyst inhibitor, from gaseous olefin containing streams which contain CO_2 at low level concentrations (i.e. down to 1 ppm).

Molecular sieves are used as adsorbents for CO_2 but are inefficient when used for the removal of CO_2 from a gaseous stream containing low molecular weight olefins such as ethylene.

Caustic scrubbers or bulk caustic scrubbers can function as adsorbents for CO_2 from a gaseous stream but have the disadvantages of being hazardous, non-regenerable, and adding water to the stream.

Therefore, an object of this invention is to provide a process for the removal of CO_2 from gaseous streams employing a regenerable adsorbent which removes even small amounts of CO_2 .

Other aspects, objects, and the several advantages of the present invention are apparent from a study of this disclosure and the appended claims.

In accordance with the present invention, we have discovered that CO_2 can be efficiently removed from a gaseous stream containing a C_2 to C_4 olefin or mixtures thereof by contacting the stream with a regenerable adsorbent consisting essentially of a calcined alkali metal compound-treated alumina as defined in the claims. The removal of water, achieved by the use of alumina in general as known to one skilled in the art, is also within the scope of the present invention. Preferred embodiments of the invention are defined in the dependent claims.

Although it is well known in the art to remove CO_2 from gaseous streams by means of solid caustic or caustic solutions (as indicated earlier in this disclosure) which convert CO_2 to an alkali metal carbonate such as Na_2CO_3 , this does not occur in the present invention. Instead, CO_2 is chemisorbed on an alkali metal oxide (e.g. Na_2O) alumina surface and can be readily desorbed by mild heating and sweeping the adsorbent bed with a gas free of CO_2 .

The present invention is not to be confused with the use of chemical processes which convert CO_2 to alkali metal carbonates in a non-regenerable process which adds water to the gas stream being processed (i.e. $2 NaOH + CO_2 \rightarrow Na_2CO_3 + H_2O$). In the process of the present invention, CO_2 is removed by a regenerable alumina which at the same time can be used to remove traces of water from the process gas.

The gaseous stream treated in accordance with the present invention will contain a C_2 to C_4 olefin or mixtures thereof. Such olefins include ethylene, propylene, isobutylene, 1-butene and 2-butene.

The alumina employed in the present invention can be any alumina known in the art. Gamma and eta alumina are preferred and are typically employed.

Preferably the alumina employed will contain less than about 20 percent impurities such as SiO_2 , Na_2O , Fe_2O_3 , TiO_2 and SO_2 .

Broadly the surface area of the alumina should be at least $50 m^2/g$ or greater and preferably $200 m^2/g$ or greater.

The alumina employed should contain from 1 to 6 weight percent Group IA alkali metal calculated as the oxide form. The level of alkali metal treatment will vary based on the particular alkali metal employed. Thus, when the alumina is treated with a lithium compound, the preferred level of lithium contacted with the alumina support is about 1 to about 3 weight percent calculated as Li_2O . When sodium is employed as the alkali metal, the range of about 2 to about 4 weight percent Na_2O is preferred. When potassium is employed as the alkali metal, the treatment range of about 3 to about 6 weight percent K_2O is preferred.

Typically the calcined alkali metal compound-treated alumina is prepared by contacting the alumina with a Group IA alkali metal compound dissolved in a suitable solvent such as water, alcohols, and esters. Water is preferred. Alkali metal compound-alumina contacting can be carried out in any suitable manner known in the art for solid-liquid contacting. For example, the techniques of incipient wetness or bulk solution can be employed. After sufficient contacting of the alumina and alkali metal compound containing solution, excess solution, if any, is removed by filtration, or decantation. Then the treated alumina is heated to a temperature from about $150^\circ C$ to about $700^\circ C$, preferably about $200^\circ C$ to about $400^\circ C$. Heating is maintained for about 1—24 hours, preferably about 5—10 hours under a gas flow of about 100—1000 GHSV (gaseous hourly space velocity). Suitable gases include N_2 , air, natural gas stream, argon, and helium. Caution should be used with carbonaceous streams such as a natural gas stream to maintain the calcination temperature below about $300^\circ C$ to minimize formation of carbon deposits on the catalyst. When an oxygen containing gas is employed during catalyst preparation, it is desirable to purge the finished catalyst with an inert gas such as N_2 , Ar, He, to ensure that oxygen is not introduced into the hydrocarbon feed stream. Although pressure is not critical, it is preferred to maintain the pressure during catalyst calcination near atmospheric or below to aid removal of gaseous species generated during the heating process.

Alkali metal compounds suitable for use in the present invention are soluble in the solvents disclosed

previously, and are convertible under calcination conditions to the alkali metal oxide form, M_2O . Examples of suitable compounds include the hydroxide, nitrate, and acetate forms of the alkali metals. Hydroxides of lithium, sodium, and potassium are preferred because of their availability and cost.

The rate at which the olefin stream is fed to the adsorbent bed is not critical but will vary with reactor size. Broadly, the rate of olefin introduction will be about 1—15 WHSV (weight hourly space velocity), preferably about 5—8 WHSV. For a gaseous feed such as ethylene, the WHSV will vary as a function of the pressure employed. In any event, it should be at a rate sufficient to effect efficient contact between the feed and the treated alumina.

This invention is well suited for continuous reaction in which the olefin feed is continuously passed over a bed of calcined alkali metal compound-treated alumina at the desired reaction conditions.

Generally, the process of the present invention is carried out at a temperature of from about 0°C to about 100°C. Presently preferred is a temperature in the range of from about 25°C to about 50°C.

The pressure under which this invention is practiced will be from about atmospheric to about 1500 psig. Preferably, the pressure will be from about 400 to about 1000 psig (2750 to about 6900 kPa) with ethylene; atmospheric to about 130 psig (900 kPa) with propylene; and atmospheric to about 30 psig (206 kPa) for a butene stream.

In light of the effectiveness of the present invention, olefin streams containing as little as 1 ppm of an inhibitor such as CO_2 can be processed.

The calcined alkali metal compound-treated alumina may readily be regenerated by heating it in the presence of a gas stream such as N_2 , He, Ar, air, or a natural gas stream. As described above, it is desirable to purge the regenerated catalyst bed with an inert gas such as N_2 , He, or Ar prior to returning the catalyst to the process when an oxygen containing stream is used for catalyst regeneration.

A temperature of from about 150°C to about 700°C, preferably about 200°C to about 400°C is employed until no further CO_2 and water vapor removal from the catalyst is achieved. Typically, regeneration takes about 1 to 24 hours, preferably 5—10 hours.

For typical plant scale operation, the inventive catalyst could be deployed in three packed beds such that at any given time one bed would be undergoing regeneration, and the other two beds in series would be contacting the olefin containing feed stream. As the first bed to contact the olefin containing feed stream becomes saturated with CO_2 (as evidenced by CO_2 in the effluent from the first bed), the backup catalyst bed would become the primary bed for contacting the olefin containing feed stream; the freshly regenerated bed would be put on stream as the backup catalyst bed, and the saturated bed would be treated under suitable regeneration conditions.

The following examples further illustrate the present invention.

Examples

Example I

Alumina treatment

Adsorbents from a variety of supplies were employed:

Supplier	Support designation
Alcoa	H-151 Al_2O_3
Alcoa	Z-100 Al_2O_3
Kaiser	K-201 Al_2O_3
Philadelphia Quartz Corp.	PQ 108 SiO_2
Davison	Grade 979 SiO_2/Al_2O_3
Linde	5A molecular sieve
Linde	13X molecular sieve

The general procedure employed for treating alumina supports with an alkali metal compound was as follows. All alumina supports employed were crushed and sieved before use, 10—28 mesh (3—10 mesh per cm) particles being collected for use in the laboratory adsorption studies. The desired amount of hydroxide, such as sodium hydroxide, was dissolved in a volume of water just sufficient to wet the alumina thoroughly. The alumina adsorbent to be employed was then added to the alkali metal compound containing solution. The wet alumina support was then dried, such as by heating to above 100°C for about 20 minutes or longer. Dry, alkali metal hydroxide treated adsorbent was typically activated by heating to about 260°C for 5 hours in N_2 with flow maintained at about 40 l/hr.

For example, alumina adsorbent treated with enough sodium hydroxide to give 2.0% Na_2O was prepared as follows. NaOH in the amount of 0.21 g was dissolved in 5 ml of water. To this solution, 8.0 g of Alcoa H-151 alumina was added with stirring. The wet alumina was heated in an evaporating dish to remove most of the excess water and then was activated at 260°C for 5 hrs. under a 40 l/hr. nitrogen flow.

Example II

In a series of tests, 50—60 mg of activated adsorbent was placed on the pan of a Perkin-Elmer TGS-2 thermogravimetric analyzer (TGA), heated to 260°C and held at 260°C while being swept with helium at 1.5

SCFM (42.5 ltr/min standard condition) until a constant weight was reached (indicating loss of moisture had ceased). The sample holder was then cooled to 30°C in the helium stream and the sample weight was recorded. A helium-CO₂ mixture containing 10 mole percent CO₂ was then substituted for the helium stream, and the gain in weight due to CO₂ adsorption was followed until a constant weight was reached, usually within 30 minutes or less. The weight gain was recorded and the weight percent CO₂ loading was calculated. In some cases the adsorbent was regenerated by heating to 260°C in helium and holding at 260°C until weight loss ceased. The adsorbent was then recooled and the CO₂ adsorption experiment repeated. Results of these tests are shown in Table I.

TABLE I

10	Description of adsorbent	Promoter		% CO ₂ loading
		Ion	Concentration, as M ₂ O, %	
			Added ~Total	
15	H-151 Alumina	Na	0 2.7	2.0
20	H-151 Alumina	Na	1.4 4.1	2.3
	H-151 Alumina	Na	2.0 4.7	2.7
	H-151 Alumina	Na	2.8 5.5	2.9
25	K-201 Alumina	Na	0 0.3	1.6
	K-201 Alumina	Na	2.8 3.1	2.9
30	K-201 Alumina	Na	4.2 4.5	3.4
	K-201 Alumina*	Na	4.2 4.5	3.2
35	K-201 Alumina	Na	5.6 5.9	3.4
	K-201 Alumina	Li	1.3 1.6	2.2
40	K-201 Alumina	K	3.2 3.5	2.4
	K-201 Alumina	K	4.2 4.5	2.9
45	5A Mole sieve	—	— —	12.2
	13X Mole sieve	—	— —	12.1
50	PQ 108 Silica	Na	0 0.1	0.4
	Davison SiO ₂ /Al ₂ O ₃	Na	2.8 2.9	0.6
55	NaOH	—	— —	12.5

* After regeneration of adsorbent from previous run.

The data show that CO₂ adsorption capacity increased sharply when Na₂O content of alumina was increased from sample to sample. The H-151 alumina, which originally contained about 2.7% Na₂O as an impurity, and the K-201 alumina, which originally contained about 0.3% Na₂O, were almost equally effective in CO₂ adsorption when promoted with additional Na₂O. The Li₂O and K₂O promoted aluminas were also effective in adsorbing CO₂. In these experiments, the molecular sieves were quite effective in adsorbing CO₂ from helium, but were inefficient in the presence of ethylene, as shown in Example III. Silica and silica-alumina were less efficient than alumina. Bulk NaOH removed CO₂ but could not be regenerated.

Example III

A series of tests, designed to closely simulate conditions used in treating ethylene monomer with alumina in a polyethylene plant were conducted as follows. A 2—3 g sample of adsorbent to be tested, previously activated at 260°C, was transferred into a 1/8"×2 1/2" (3 mm×64 mm) brass pipe closed at each end with a needle valve. Ethylene, containing 11—17 ppm CO₂ (by volume) was passed through the adsorbent maintained at 700 psig (4800 kPa) at a rate of approximately 7 g per hour per g of adsorbent. The effluent ethylene was periodically sampled and analyzed for CO₂ as follows: The sample was injected into a Carle Model 211M chromatograph equipped with a 3/16"×22' (5 mm×560 mm) Carbopack B column (80/100 mesh (34/41 mesh per cm)) operated at 50°C. A 60 ml/min He carrier gas stream carried the sample through the column and into a methanizer unit which converted the CO₂ to methane before being determined by a flame ionization detector. Sensitivity for CO₂ was less than 1 ppm. The ethylene flow and sampling were continued several days as necessary until CO₂ broke through the adsorbent and until the CO₂ concentration in the effluent approached that in the feed. The CO₂ concentration in the effluent was plotted against time, and the time to breakthrough and saturation determined. The total amount of CO₂ adsorbed was calculated and expressed as weight percent CO₂ loading on the adsorbent. Results are shown in Table II.

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TABLE II

Run No.	Description of adsorbent	Promoter			Conc. as M ₂ O%		Catalyst, g	Ethylene feed ¹		Break-through, hours	Saturation, hours	%CO ₂ loading
		Ion	Added		~Total	Flow, l/h.		CO ₂ (ppm)				
1	H-151 Alumina	Na	0	2.7	2.7	2.7	20	11	26	63	0.7	
2	H-151 Alumina	Na	1.0	3.7	2.6	15	15	15	41	88	1.1	
3	H-151 Alumina	Na	1.4	4.1	2.1	12	15	15	48	78	1.2	
4	H-151 Alumina	Na	2.0	4.7	2.8	16	14	14	65	107	1.3	
5	H-151 Alumina ⁽¹⁾	Na	2.0	4.7	2.8	15	13	13	65	104	1.2	
6	H-151 Alumina	Na	2.8	5.5	2.8	14	13	13	83	139	1.4	
7	K-201 Alumina	Na	0	0.3	1.6	9	13	13	37	58	0.7	
8	K-201 Alumina	Na	1.0	1.3	1.8	10	17	17	44	75	1.1	
9	K-201 Alumina	Na	2.0	2.3	2.2	12	13	13	115	146	1.8	
10	K-201 Alumina	Na	2.8	3.1	2.0	11	15	15	88	128	1.7	
11	K-201 Alumina ⁽²⁾	Na	2.8	3.1	2.0	11	15	15	102	132	1.9	
12	K-201 Alumina ⁽²⁾	Na	2.8	3.1	2.0	11	14	14	94	123	1.7	
13	K-201 Alumina	Na	4.2	4.5	2.0	11	15	15	91	122	1.7	
14	K-201 Alumina	Li	2.7	3.0	1.9	11	14	14	87	144	1.8	
15	Z-100 Alumina	—	—	—	2.4	13.5	14	14	42	75	0.9	
16	KOH	—	—	—	3.0	17	14	14	31	67	1.0	
17	5A Mole sieve	—	—	—	2.3	20	13	13	~3	~18	0.2	
18	13X Mole sieve	—	—	—	2.0	11	13	13	~3	~18	0.2	

⁽¹⁾ Regeneration of catalyst from Run 4. ⁽²⁾ Repeated regenerations of catalyst from Run 10.

From Table II it can be seen that sodium oxide promoted aluminas were far more effective than molecular sieves, which are frequently recommended by the adsorbent industry for CO₂ removal from gas streams. Li₂O on alumina was also effective. KOH pellets, normally considered preferable to NaOH when used in bulk for CO₂ removal, were ineffective under these conditions (dry).

5 The optimum Na₂O concentration proved to be in the range of 2—4 weight percent of the alumina.

Example IV

In a large polyethylene plant, alumina beds, used primarily to remove traces of moisture from ethylene at 700 psig (4800 kPa) and a flow ratio of 5—10 kg ethylene per hour per kg of alumina, had to be
10 regenerated about every 4 days to effect CO₂ removal when present at 5—6 ppm. The alumina was then removed and replaced with identical alumina to which had been added 3 weight percent Na₂O, added as a NaOH solution. At the same CO₂ concentration of 5—6 ppm in the ethylene, regeneration was now required only every 2 weeks.

This example shows that a calcined alkali metal compound treated alumina is considerably more
15 effective for CO₂ removal than untreated alumina.

Claims

1. A process for removing low concentrations of CO₂ from a gaseous olefin stream containing at least
20 one C₂ to C₄ olefin characterized by passing said gaseous olefin stream containing CO₂ into contact with a regenerable, calcined adsorbent comprising from 1 to 6 weight percent of an oxide of an alkali metal selected from sodium, potassium and lithium on alumina, said regenerable, calcined adsorbent having been made by (a) contacting alumina with an alkali metal compound selected from sodium, potassium and lithium compounds which are convertible to the metal oxide on calcination, (b) drying the resulting alkali
25 metal compound-treated alumina, and (c) thereafter calcining the dried treated alumina.

2. The process of claim 1 characterized in that said olefin stream is ethylene and said alkali metal compound is selected from hydroxides, nitrates and acetates of sodium, potassium and lithium.

3. The process of claim 2 characterized in that said alkali metal compound is selected from lithium hydroxide, sodium hydroxide and potassium hydroxide.

30 4. The process of any of the preceding claims characterized in that said alkali metal oxide is lithium oxide which is present in an amount from 1 to 3 weight percent.

5. The process of any of claims 1 to 3 characterized in that said alkali metal oxide is sodium oxide which is present in an amount from 2 to 4 weight percent.

35 6. The process of any of claims 1 to 3 characterized in that said alkali metal oxide is potassium oxide which is present in an amount from 3 to 6 weight percent.

Patentansprüche

1. Verfahren zum Entfernen niedriger CO₂-Konzentrationen aus einem gasförmigen Olefinstrom, der
40 zumindest ein C₂- bis C₄-Olefin enthält, dadurch gekennzeichnet, daß man den gasförmigen, CO₂ enthaltenden Olefinstrom in Kontakt bringt mit einem regenerierbaren, calcinierten Adsorptionsmittel, das von 1 bis 6 Gew.% eines Oxids eines Alkalimetalls enthält, das aus Natrium, Kalium und Lithium auf Aluminiumoxid ausgewählt wird, wobei das regenerierbare, calcinierte Adsorptionsmittel hergestellt wurde durch (a) Kontaktieren von Aluminiumoxid mit einer Alkalimetallverbindung, die aus Natrium-,
45 Kalium-, und Lithiumverbindungen ausgewählt werden, die durch Calcinierung in das Metalloxid überführbar sind, (b) Trocknen des entstehenden, mit der Alkalimetallverbindung behandelten Aluminiumoxids und (c) danach Calcinieren des getrockneten behandelten Aluminiumoxids.

2. Verfahren nach Anspruch 1, dadurch gekennzeichnet, daß der Olefinstrom Ethylen ist und daß die Alkalimetallverbindung aus Hydroxiden, Nitraten und Acetaten von Natrium, Kalium und Lithium
50 ausgewählt wird.

3. Verfahren nach Anspruch 2, dadurch gekennzeichnet, daß die Alkalimetallverbindung aus Lithiumhydroxid, Natriumhydroxid und Kaliumhydroxid ausgewählt wird.

4. Verfahren nach einem der vorhergehenden Ansprüche, dadurch gekennzeichnet, daß das Alkalimetallloxid Lithiumoxid ist, welches in einer Menge von 1 bis 3 Gew.% anwesend ist.

55 5. Verfahren nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß das Alkalimetallloxid Natriumoxid ist, das in einer Menge von 2 bis 4 Gew.% anwesend ist.

6. Verfahren nach einem der Ansprüche 1 bis 3, dadurch gekennzeichnet, daß das Alkalimetallloxid Kaliumoxid ist, das in einer Menge von 3 bis 6 Gew.% anwesend ist.

60 Revendications

1. Procédé pour éliminer de faibles concentrations de CO₂ d'un courant oléfinique gazeux contenant au moins une oléfine en C₂ à C₄, caractérisé en ce que l'on fait passer le courant oléfinique gazeux contenant du CO₂ au contact d'un adsorbant calciné, régénérable, contenant de 1 à 6 pourcent en poids d'un oxyde
65 d'un métal alcalin choisi parmi le sodium, le potassium et le lithium sur de l'alumine, l'adsorbant calciné.

From Table II it can be seen that sodium oxide promoted aluminas were far more effective than molecular sieves, which are frequently recommended by the adsorbent industry for CO₂ removal from gas streams. Li₂O on alumina was also effective. KOH pellets, normally considered preferable to NaOH when used in bulk for CO₂ removal, were ineffective under these conditions (dry).

5 The optimum Na₂O concentration proved to be in the range of 2—4 weight percent of the alumina.

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In a large polyethylene plant, alumina beds, used primarily to remove traces of moisture from ethylene at 700 psig (4800 kPa) and a flow ratio of 5—10 kg ethylene per hour per kg of alumina, had to be
10 regenerated about every 4 days to effect CO₂ removal when present at 5—6 ppm. The alumina was then removed and replaced with identical alumina to which had been added 3 weight percent Na₂O, added as a NaOH solution. At the same CO₂ concentration of 5—6 ppm in the ethylene, regeneration was now required only every 2 weeks.

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25 metal compound-treated alumina, and (c) thereafter calcining the dried treated alumina.

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45 Kalium-, und Lithiumverbindungen ausgewählt werden, die durch Calcinierung in das Metalloxid überführbar sind, (b) Trocknen des entstehenden, mit der Alkalimetallverbindung behandelten Aluminiumoxids und (c) danach Calcinieren des getrockneten behandelten Aluminiumoxids.

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60 Revendications

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moins un oléfine en C₂ à C₄, caractérisé en ce que l'on fait passer le courant oléfinique gazeux contenant
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65 d'un métal alcalin choisi parmi le sodium, le potassium et le lithium sur de l'alumine, l'adsorbant calciné.



régénérable, ayant été préparé par (a) mise en contact d'alumine avec un composé de métal alcalin choisi parmi les composés du sodium, du potassium et du lithium qui sont convertibles en l'oxyde de métal par calcination, (b) séchage de l'alumine traitée par le composé de métal alcalin ainsi obtenue, et (c) calcination subséquente de l'alumine traitée et séchée.

5 2. Procédé suivant la revendication 1, caractérisé en ce que le courant oléfinique précité est constitué d'éthylène et le composé de métal alcalin en question est choisi parmi les hydroxydes, nitrates et acétates de sodium, de potassium et de lithium.

3. Procédé suivant la revendication 2, caractérisé en ce que l'on choisit le composé de métal alcalin précité parmi l'hydroxyde de lithium, l'hydroxyde de sodium et l'hydroxyde de potassium.

10 4. Procédé suivant l'une quelconque des revendications précédentes, caractérisé en ce que l'oxyde de métal alcalin en question est l'oxyde de lithium qui est présent en une quantité de 1 à 3 pourcent en poids.

5. Procédé suivant l'une quelconque des revendications 1 à 3, caractérisé en ce que l'oxyde de métal alcalin en question est l'oxyde de sodium qui est présent en une quantité de 2 à 4 pourcent en poids.

15 6. Procédé suivant l'une quelconque des revendications 1 à 3, caractérisé en ce que l'oxyde de métal alcalin en question est l'oxyde de potassium qui est présent en une quantité de 3 à 6 pourcent en poids.

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