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División de Nuevas Creaciones

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SOLICITUD

# PATENTE DE INVENCION

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54. **TITULO** Tratamiento de efluentes cáusticos de refinería consumidos

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71. **SOLICITANTE** Emco/Dayton Inc

**DOMICILIO** Ohio, Estados unidos

74. **APODERADO** Dilia María Rodríguez d' Aleman

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**1) SOLICITUD DE:**

☒ Patente de invención ☐ Patente de Modelo de Utilidad

☐ Capitulo I. ☒ Capitulo II.

| | | |
|---|---|---|
| **2) SOLICITANTE (71)** | **Nombre:, EMCON/OWT, INC.**<br>**Dirección: 6910 Treeline Drive, Suite F, Brecksville, OH 44141 Estados Unidos**<br>**Nacionalidad o domicilio: Brecksville, OH Estados Unidos**<br>**Lugar de constitución: Ohio, Estados Unidos** | **IDENTIFICACION**<br>**C.C.** ☐ **NIT:** ☐<br>**C.E.** ☐ **OTRO** ☐<br>**Cual** _____<br>**NÚMERO** _____ |
| | **Teléfono:** **Fax:**<br>**E. Mail:** | |
| **3) REPRESENTANTE O APODERADO** | **Nombre:** DILIA MARÍA RODRÍGUEZ D'ALEMAN<br>**Dirección:** Carrera 11 N°. 93-43 Of. 404 Bogotá D.C.<br>**Teléfono:** 6350835 **Fax:** 6350824<br>**E. Mail:** clarkemodet@clarkemodet.com.co | **IDENTIFICACIÓN**<br>**C.C.** ☒ **NIT:** ☐<br>**C.E.** ☐ **OTRO** ☐<br>**NÚMERO:** 41.654.882<br>**TP:** 20824 CSJ |
| **4) INVENTOR (ES) (72)** | **Nombre: 1. DUESEL, Bernard, F., Jr.; 2. GIBBONS, John, P.; 3. RUTSCH, Michael, J.**<br>**Dirección: 1. 4 Partridge Lane, Goshen, NY 10924, Estados Unidos; 2. 4 Harold Avenue, Cornwall, NY 12518; 3. 8329 South 75th East Avenue, Tulsa, OK 74133, Estados Unidos**<br>**Nacionalidad o Domicilio: Todos los anteriores, Estadounidenses** | |

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**10) ANEXOS**

- [x] Comprobante de pago de la tasa de presentación de la solicitud
- [ ] Comprobante de pago por reivindicación de prioridad
- [ ] Documento que acredita la existencia y representación legal cuando el solicitante sea persona Jurídica
- [ ] Poderes, si fuera el caso
- [ ] Documento de cesión del inventor al solicitante o a su causante
- [ ] Declaraciones
- [x] Copia de la solicitud Internacional. (Resumen, descripción, reivindicación, Dibujos).
- [x] *Traducción de la solicitud internacional al castellano.* (Resumen, descripción, reivindicación, Dibujos)
- [ ] Copia del examen preliminar efectuado por la IPEA – Opinión escrita
- [ ] De ser el caso, copia del contrato de acceso
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- [ ] De ser el caso, certificado de depósito del material biológico
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- [ ] De ser el caso, información sobre otras solicitudes de patente o títulos obtenidos en el extranjero por el mismo titular o su causante, relacionados parcial o totalmente con la invención de esta solicitud.
- [x] Otros.
  1. Reporte internacional de búsqueda
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**11) FIGURA CARACTERÍSTICA:**

**12) Solicitud concesión de la patente,**

**NOMBRE:** DILIA RODRÍGUEZ D´ALEMAN

**FIRMA:**

**C.C.** 41.654.882 de Bogota **TP** 20824 CSJ

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(71) Applicant *(for all designated States except US)*: **EMCON/OWT, INC.** [US/US]; 6910 Treeline Drive, Suite F, Brecksville, OH 44141 (US).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: **DUESEL, Bernard, F., Jr.** [US/US]; 4 Partridge Lane, Goshen, NY 10924 (US). **GIBBONS, John, P.** [US/US]; 4 Harold Avenue, Cornwall, NY 12518 (US). **RUTSCH, Michael, J.** [US/US]; 8329 South 75th East Avenue, Tulsa, OK 74133 (US).

(74) Agent: **BERKLEY, Richard, G.**, Baker Botts L.L.P., 30 Rockefeller Plaza, New York, NY 10112-4498 (US).

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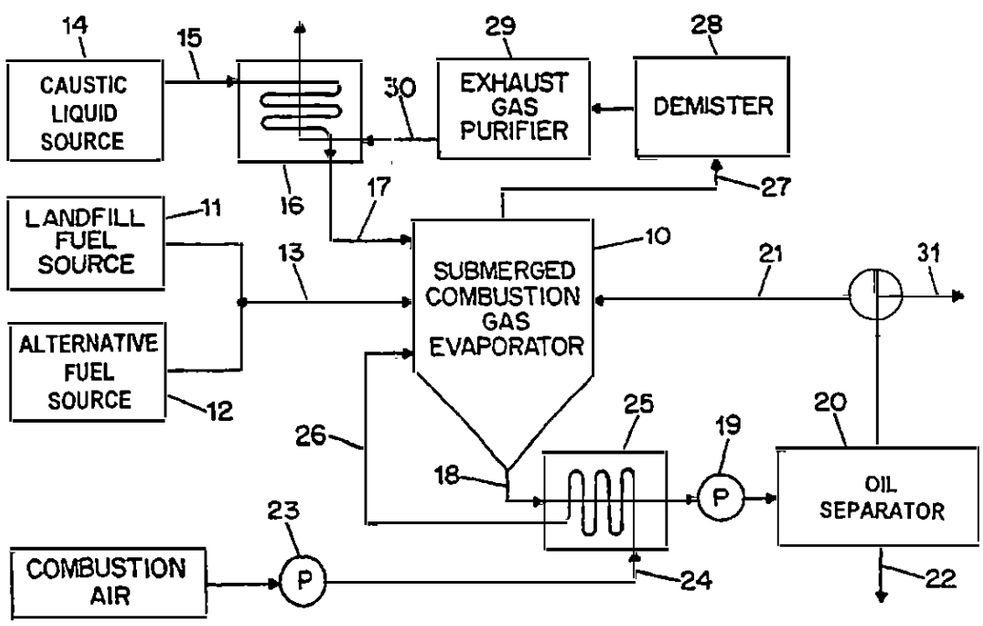
(54) Title: TREATMENT OF SPENT CAUSTIC REFINERY EFFLUENTS




(57) Abstract: In the methods for treatment of caustic effluents described in the specification, a spent caustic refinery effluent is supplied to a submerged combustion gas evaporator in which hot combustion gas containing carbon dioxide is injected into the caustic liquid to concentrate the liquid and convert a hydroxide constituent to a carbonate. Where the caustic effluent is from a petroleum refinery, oil in the waste liquid is separated from the aqueous constituent before, during or after concentration.

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# TREATMENT OF SPENT CAUSTIC REFINERY EFFLUENTS

## SPECIFICATION

## RELATED APPLICATION

This application claims priority to United States Patent Application No. 10/234,559, which was filed on September 4, 2002, and is incorporated herewith by reference.

## BACKGROUND OF THE INVENTION

This invention relates to processes for treating spent caustic effluents, for example, effluents from petroleum refineries, aluminum manufacturing, food processing or the like.

Many industrial operations generate dilute spent caustic effluents such as petroleum refining processes. A typical dilute spent caustic effluent from such refineries may contain about 5% to 12% sodium hydroxide (w/w) with varying but significant quantities of organic compounds that include a range of mercaptans, sulfidic oils, naphthenic acids, cresylic acids and derivatives. Included in the mixture are lesser quantities of other inorganic and organic compounds. In addition, a petroleum refinery effluent may contain approximately 5% to 20% by volume of oil along with the aqueous caustic solution. Traditionally such effluents have been considered essentially useless waste streams but have required significant processing before disposal to avoid contamination of the environment.

The patent to Helleur No. 4,079,585 describes a method and apparatus for removal and recovery of a constituent from industrial and municipal waste streams by evaporation and concentration of the constituent as a result of intimate and turbulent contact between the waste stream liquid and hot gases produced by combustion in a scrubbing tower and states that submerged combustion techniques can also be used to advantage in the process. According to that patent undesirable volatile pollutants can be removed without vaporizing them by addition of an alkali such as lime or caustic to retain acidic volatile pollutants such as sulfur dioxide, hydrogen sulfide, etc. in solution so that they can be disposed of in liquid form. The

Helleur patent describes the process as being applicable to spent material from such industries as the oil industry and notes that, to avoid vaporization of selected combustible volatiles, the temperature of the combustion gases should be maintain below the flash point of the volatiles by cooling the combustion gas before contact with the liquid.

The Young et al. Patent No. 4,16,028 discloses a submerged combustion evaporator as the first stage in a process for concentration of constituents of industrial waste streams.

In the Ohkawa et al. Patent No. 3,966,594, treatment of waste water containing water-soluble organic substances in various ways is described and the submerged combustion method is stated to be industrially insufficient in terms of concentration and combustion. Instead, that patent describes a process in which the waste water is treated with a water-insoluble organic solvent solution of an organic constituent.

According to the Anderson Patent No. 4,188,291 industrial waste water is processed by a submerged combustion evaporator and carbon dioxide in the combustion gases supplied to the waste water is sequestered by calcium hydroxide which has been added to produce calcium carbonate which is then separated from the waste stream.

The spent caustic treatment process described in the DeRoeck et al. Patent No. 5,244,576 introduces refinery gases containing carbon dioxide and hydrogen sulfide into a sodium hydroxide solution to convert the carbon dioxide to sodium carbonate.

In the Connally Patent No. 3,985,609 concentration of constituents in a liquid to be concentrated is effected by supplying the liquid to a submerged combustion evaporator.

The Echols Patent No. 5,934,207 describes evaporation of leachate by directing flames from a burner to which landfill gas is supplied into a fire tube heater immersed in a tank containing leachate so as to heat and vaporize the liquid constituents in the leachate while disposing of the landfill gas.

*For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.*

The Duesel Patent No. 5,342,482 discloses the use of landfill gas as a fuel source for a submerged combustion gas evaporator in a leachate evaporation process.

## SUMMARY OF THE INVENTION

Accordingly, it is an object of the present invention to provide a process for treating spent caustic refinery effluents which overcomes disadvantages of the prior art.

Another object of the invention is to provide a process for treating caustic effluents from a refinery to convert a caustic constituent to a useful commercial product.

A further object of the invention is to provide a process for treating petroleum refinery effluents to produce a reusable oil product as well as a converted caustic product.

An additional object to the invention is to provide a process for treating caustic effluents utilizing carbon dioxide in a combustion gas supplied to the effluent to convert a caustic constituent to a carbonate.

These and other objects of the invention are attained by supplying a caustic effluent to a submerged combustion gas evaporator to which combustion gases are supplied at a temperature and quantity selected to remove undesired vapor and gases while retaining a desired liquid constituent of the effluent, and separating the desired constituent from the remaining constituents of the effluent. In a particular embodiment of the invention the caustic effluent is a petroleum refinery effluent and carbon dioxide in the combustion gas supplied to the effluent converts a caustic constituent in the effluent to a carbonate such as sodium carbonate. In addition, oil is separated from the other effluent constituents, before, during or after evaporation, for reuse or further refining.

According to another aspect of the invention, at least some of the fuel used to supply combustion gas to the submerged combustion gas evaporator is landfill gas and the carbon dioxide content of the combustion gas is used to convert the

caustic constituent of the effluent to a carbonate, thereby preventing dissemination of the carbon dioxide into the atmosphere.

In a representative embodiment of the invention the spent effluent supplied to the submerged combustion gas evaporator has an equivalent sodium hydroxide content of about 5% to 12% weight percent and an oil content of about 5% to 20% by volume and the aqueous content of the spent effluent is reduced by evaporation as required to produce a desired carbonate concentration. The concentrated liquid may contain about 20% to 30% sodium carbonate and the oil constituent is separated for reuse after concentration by a gravity separator.

## BRIEF DESCRIPTION OF THE DRAWINGS

Further objects and advantages of the invention will be apparent from a reading of the following description in conjunction with the accompanied drawings in which:

Fig. 1 is a schematic block diagram illustrating a representative embodiment of an arrangement for carrying out a process for treating caustic effluents in accordance with the invention;

Fig. 2 is a schematic sectional view illustrating a representative embodiment of a submerged combustion gas evaporator for use in carrying out the process of the invention; and

Fig. 3 is a schematic block diagram illustrating a further representative embodiment of an arrangement for treating caustic effluents in accordance with the invention.

## DESCRIPTION OF PREFERRED EMBODIMENTS

In the representative arrangement for carrying out the process of the invention schematically illustrated in Fig. 1, a submerged combustion gas evaporator 10, which is described in more detail in connection with Fig 2, receives combustible gas from a landfill or other biogas source 11 or from an alternative fuel source 12 providing natural gas, propane, butane or the like or providing a liquid fuel such as waste petroleum oils or the like or from both. The evaporator 10 may be operated on a batch, batch-continuous, or continuous basis and the fuel gas from the sources 11

and 12 may be collected and stored for use or be supplied continuously in accordance with the needs of the evaporator. In one embodiment the system is located closely adjacent to a landfill from which landfill gases are conveniently available, but, if desired, landfill gases may be piped or transported from one or more landfills at remote locations to processing stations adjacent to a source of caustic effluent liquid such as a refinery or other manufacturing facility.

In the embodiment illustrated in Fig. 1 a caustic liquid from a source 14 such as spent caustic effluent from a petroleum refinery, aluminum manufacturing plant or food processing facility is transmitted through a line 15 to a heat exchange unit 16 for preheating by hot exhaust gases and then piped through a line 17 to the inlet to the submerged combustion gas evaporator 10. The caustic in the spent effluent may be sodium hydroxide or any other alkaline material and the spent effluent may be from a petroleum refinery, aluminum manufacturing plant, food processing facility or from various industrial operations such as scrubbing. The sodium hydroxide equivalency of the caustic material in the spent effluent from the source 14 is preferably in a range from about 1% to about 50%, desirably in a range from about 1% to 15% and most desirably in a range from about 2% to 12%. If the spent effluent is from a petroleum refinery and contains a substantial proportion of oil, the spent effluent may be passed through a preliminary oil and water separator (not shown) to remove at least a portion of the oil before it is delivered to the evaporator 10.

Following evaporation of vaporizable constituents from the spent effluent in the evaporator 10, concentrated liquid is removed through an outlet 18 and transmitted by a pump 19 through an oil separator 20 to remove oil from the aqueous constituents of the concentrated liquid which pass from the separator to an outlet 31. If necessary, some or all of the aqueous constituents can be recycled through a line 21 back to the evaporator 10 for further processing after separation of the oil. The separated oil is removed from the separator through a line 22 for sale, further processing or recycle. The oil separator 20 may be any conventional immiscible liquid separator such as a gravity settling tank, an inclined plate separator or a centrifuge.

Combustion air driven by a blower 23 and supplied through a line 24 is preheated by circulation through a heat exchange unit 25 through which the concentrated liquid passes from the outlet 18 and the preheated combustion air is supplied to the evaporator 10 through a line 26. The heat exchange in the unit 25 may be used to improve the efficiency of the burner. If desired some of the combustion air from the blower 23 may be diverted to an in-line air stripper (not shown) in the line 17 to remove volatile compounds from the spent effluent being supplied to the evaporator and supply them with the combustion air to the burner in the evaporator 10. Exhaust gases are withdrawn from the evaporator 10 through a line 27 and then passed through a demister 28 in which entrained liquid constituents are removed, after which the gas is passed through an exhaust purification unit 29 such as an enclosed flare. In the purification unit 29 volatile organic compounds and other exhaust gas constituents are thermally oxidized or otherwise treated to render them odorless and harmless before being released into the atmosphere through a line 30 which passes through the heat exchange unit 16.

The arrangement of a preferred form of submerged combustion gas evaporator for use in the process of the invention is illustrated in Fig. 2. As shown in that figure the evaporator 10 has a housing 40 with a downwardly converging conical bottom 41 leading to a discharge valve 42 through which the concentrated aqueous caustic liquid and oil, if present, pass into the discharge line 18. Within the housing 40 liquids and entrained solids, if any, can settle and be directed by the conical housing portion 41 toward the discharge valve 42. At the top of the housing 40 a cover 47 is sealed with a sealing gasket 48 to the housing wall and a burner 49 is mounted to the cover.

Combustion gases from the burner 49 are directed downwardly through a flame and combustion gas downcomer 51 into a distributor 52 having a series of mixing sparge pipes 53. The distributor pipes have orifices 54 through which the hot combustion gases are injected into a pool 55 of the spent caustic effluent contained within the housing so as to evaporate liquid therefrom in an evaporation zone 45 by direct contact heat transfer, provide and distribute carbon dioxide to the spent effluent to effect conversion to carbonate and also drive off

vaporizable constituents. Preferably, the operating pressure within the evaporator is within a range from about 50 inches negative to 100 inches positive of water.

The upper surface 56 of the caustic waste liquid pool within the housing is maintained at a desired level by controlling the supply of spent effluent admitted into the evaporator through the line 17 from the source 14. Preferably, the level of the upper surface 56 is in a range from about 5 inches to about 100 inches above that of the distributor 52 and the temperature of the pool of spent effluent 55 is in a range from about 100°F to about 250°F. If the evaporator is being operated on a continuous basis the spent effluent is supplied to the evaporator continuously at an appropriate rate so as to maintain the surface 56 at the indicated level within the evaporator.

On the other hand, if the evaporator is being operated on a batch basis the effluent to be processed will be supplied continuously at an appropriate rate to as to maintain the surface 56 at the indicated level until the concentration of desired constituents in the effluent reaches a selected value. Thereafter the supply is terminated and the evaporator is either shut down and emptied or operation may continue until the surface of the effluent within the evaporator falls to a lower level (not shown) which is above the highest part of the gas distributor 52 at which time the evaporator will be shut down and the concentrated effluent will be discharged, after which the evaporator can be started again.

In a preferred embodiment the inlet temperature of the combustion gases in the distributor 52 is in a range from about 600°F to 1800°F, the temperature and quantity being high enough to vaporize water and volatile constituents in the spent effluent so as to concentrate desired constituents such as oil and/or a converted caustic at a desired rate.

When operated in a batch-continuous mode, the operation proceeds as in a continuous mode except that concentrated effluents are removed periodically from the discharge 42 without lowering the liquid level or shutting down the evaporator. As indicated in Fig. 1, fuel is supplied to the burner 49 through a line 13 and combustion air is supplied to the burner through a line 26 under sufficient pressure, preferably in a range from about 5 inches to about 200 inches of water, to

provide for efficient combustion and to force the combustion gases through the distributor 52 and convey the exhaust gases through the exhaust line 27.

It should be noted that, exclusive of process pumps, only a single moving member is required to carry out this process provided that a fuel supply is available at adequate pressure, i.e., the blower 23 which supplies combustion air under pressure. Accordingly, the spent caustic effluent treatment process of the invention not only removes odorous constituents from the effluent but also produces a commercially useful aqueous concentrate and separates a useful oil constituent in a simple and effective manner without the requiring complex moving parts which lead to difficulties embodied in the prior art spent caustic effluent handling systems.

Moreover, although spent caustic effluent is passed through the heat exchange units 16 and 25 in the arrangement disclosed in Fig. 1, the difficulties resulting from the prior art use of heat exchange evaporators are not encountered in those units since no evaporation is carried out in them and the consequent fouling of heat exchange surfaces is avoided. In simpler forms of spent caustic effluent treatment systems according to the invention, the heat recovery units 16 and 25 may be omitted and, if the exhaust vapor meets environmental standards for direct discharge to the atmosphere, the exhaust treatment units 28 and 29 may also be omitted.

Fig. 3 illustrates an alternative embodiment which is identical to that of Fig. 1 except that, as previously described, a source of hot gases spaced from the evaporator such as a hot gas generator 13', which may for example be an internal combustion engine, supplies hot gases through the line 13 to the hot gas distribution device 52 in a modified submerged hot gas evaporator 10', the air supply line 26 being connected to the hot gas generator 13' rather than to the evaporator 10'. In this case the hot gases from the hot gas generator are supplied to the evaporator at a pressure in a range from about 60 inches negative to about 120 inches positive of water. In all other respects the caustic liquid treatment system of Fig. 3 is the same as that shown in Fig. 1 and the evaporator 10' is the same as the evaporator 10 illustrated in Fig 2.

In accordance with the invention, spent caustic effluent from petroleum refineries or the like can be concentrated efficiently and effectively without requiring

a heat exchange evaporator of the type used in conventional spent caustic effluent treatment systems having surfaces which can be fouled by the effluent residue and therefore require periodic cleaning or replacement. Typical oil refinery spent caustic effluents which can be processed by the present invention include sulfidic, cresylic and naphthenic effluents.

As described in the paper entitled "Effluent Caustic Treating Systems Using MERICON℠ Technologies" by Merichem Chemicals and Refinery Services LLC., sulfidic caustic effluents are produced by fuel gas, liquefied petroleum gas (LPG) and gasoline treating processes. A typical refinery sulfidic caustic stream has the composition shown in Table 1 below. The usual contaminants are sodium sulfide and sodium mercaptide. These compounds cause high chemical and biological oxygen demand in the treatment process and produce odors and dangerous gases when neutralized.

Table 1

| | |
|---|---|
| Free NaOH, wt% | 2 to 10 |
| Sulfides and Bisulfides as S. wt% | 0.5 to 4 |
| Mercaptides as S, wt% | 0.1 to 4 |
| Carbonates as $CO_3$, wt% | 0 to 4 |
| pH | 13 to 14 |
| Ammonia | Trace |

Commercial uses of sulfidic caustic solutions concentrated in accordance with the invention are as treating agents in pulp and paper processing and for purifying certain metal ores in the mining industry.

Cresylic caustic effluents, which contain aromatic acid oils, are produced by caustic treating of cracked gasoline and cracked distillates. Cresylic caustic streams contain phenols, cresols and other organic acids that are generally present as water-soluble cresylates which will separate from the caustic as acid oils at a neutral pH. Cresylic caustic solutions produced from treating cracked gasolines generally come from two sources: 1) mercaptan extraction or oxidation systems using strong caustic; and 2) mercaptan oxidation systems using weak caustic. Table 2 below shows the characteristics of typical cresylic caustic effluents.

Table 2

| | Strong Caustic Operation | Dilute Caustic Operation |
|---|---|---|
| NaOH, wt% | 10 to 15 | 1 to 4 |
| Sulfides as S. wt% | 0 to 1 | 0 to 0.2 |
| Mercaptides as S, wt% | 0 to 4 | 0 to 0.5 |
| Cresylic Acids, wt% | 10 to 25 | 2 to 5 |
| Carbonates as $CO_3$, wt% | 0 to 0.5 | 0 to 0.1 |
| pH | 12 to 14 | 12 to 14 |

Concentrated cresylic caustic solutions have commercial value as chemical intermediates.

Naphthenic caustic solutions are generated from caustic treatment of kerosene and diesel cuts from a naphthenic crude slate. Table 3 below shows typical characteristics of naphthenate streams derived from kerosene and diesel stocks.

Table 3

| | Jet Fuel / Kerosene | Diesel |
|---|---|---|
| NaOH, wt% | 1 to 4 | 1 to 4 |
| Sulfides as S-, wt% | 0 to 0.1 | Trace |
| Mercaptides as S+, wt% | 0 to 0.5 | 0 to 0.5 |
| Naphthenic acids, wt% | 2 to 10 | 2 to 15 |
| Cresylics, wt% | 1 to 3 | 0 to 1 |
| pH | 12 to 14 | 12 to 14 |

Concentrated naphthenic caustic solutions have potential commercial value to processors who refine them for sale to producers of naphthenate metal salts.

Petroleum refineries typically pay costs for shipping, treatment and disposal of raw industrial waste liquids including spent caustic. The refineries would find it useful to repurchase both the caustic liquid and oil for re-use in their process if they can be deodorized and the sodium hydroxide equivalency concentrated to acceptable values. In accordance with the present invention, petroleum refinery spent caustic can be processed to satisfy those requirements effectively and efficiently.

In a pilot test of the process of the invention, a significant reduction in the level of odor in the concentrated liquid was obtained compared to the feed material. The concentrated liquid was a two-phase mixture of oil and an aqueous phase that could be readily separated by gravity separation such as decanting. After decanting, the aqueous phase assay showed that a significant concentration of caustic was achieved. The assay also showed almost 100% conversion of the sodium hydroxide in the spent caustic effluent to sodium carbonate because of the reaction of carbon dioxide in the combustion gas with the hydroxide as discussed hereinafter. In this test the concentration of sodium carbonate in the concentrated aqueous phase was approximately 30% (w/w). In this regard, the direct combustion gas sparge that takes place in the submerged combustion gas evaporator during the process causes the formation of carbonic acid in the water that is present within the evaporator during processing and carbonic acid then reacts with the caustic constituent, sodium hydroxide, to form sodium carbonate in the manner described below.

The dilute caustic feed material for the pilot test was an approximate 1:1 blend of two by-products of refinery operations known as the "sulfidic" and "cresylic" spent caustic streams. Assays of each of these streams and the material used as feed for the pilot test are presented in Table 4 below.

| TABLE 4 | | | |
|---|---|---|---|
| Composition of Constituent Feed Streams and Mix Average | | | |
| DILUTE CAUSTIC | Stream 1 | Stream 2 | Pilot Unit Feed |
| | Sulfide | Cresylate | (1:1 Mixture of Streams 1 & 2) |
| Test Results, %[1] | | | |
| | | | |
| Sodium Hydroxide | 10.82 | 10.04 | 10.43 |
| Sodium Carbonate | 2.65 | 4.14 | 3.40 |
| Sodium | 7.66 | 7.66 | 7.66 |
| CO2 | 1.1 | 1.72 | 1.41 |
| Inorganic Carbon | 0.29 | 0.47 | 0.38 |
| Hydrogen Sulfide | 0.37 | 0.15 | 0.26 |
| Total Sulfur as S | 0.62 | 1.06 | 0.84 |
| Total Carbon | 0.45 | 7.76 | 4.11 |
| Water | 86.87 | 76.12 | 81.50 |
| Total Hydrocarbon | 0 | 0 | 0 |
| Total Mercaptans | 0.12 | 0.55 | 0.34 |
| Cresols | 0.04 | 2.82 | 1.43 |
| Phenol | 0.02 | 1.31 | 0.67 |
| Alkyl Phenols | 0.03 | 2.74 | 1.39 |

[1] The total of all percentages is greater than one-hundred because some elements are double counted within various compounds.

The goal of the pilot test was to both deodorize and concentrate the sodium hydroxide content of a caustic material that was produced as a by-product in petroleum refining operations. Because the combustion gas supplied to the evaporator contained carbon dioxide, it was anticipated that some portion of the sodium hydroxide would be converted to sodium carbonate. It was not known how the

mixture of oils carried within the caustic feed material would affect, or be affected by, the process.

The results of the pilot test showed that the sodium hydroxide had been converted almost entirely to sodium carbonate, and that an oil phase was produced that was immiscible with the aqueous sodium carbonate phase. Decanting easily separated the two immiscible phases. Each of the phases produced in the experiment was judged to have significantly reduced levels of odor compared to the feed material, demonstrating that the objective of deodorizing the dilute caustic feed during the process had been achieved.

Table 5 below shows a comparison of selected constituents in the material used as feed to the pilot unit and the aqueous phase produced as a result of subjecting the material to the combined deodorization / evaporation process. In the test, the volume of the feed material was reduced to approximately one-third of the original spent caustic effluent liquid volume.

| TABLE 5 | | | |
|---|---|---|---|
| Comparison of Feed Material with Concentrate | | | |
| Aqueous Phase from First Pilot Test | Feed | Final | Comments |
| | | | |
| Test Results, %[1] | | | |
| | | | |
| Sodium Hydroxide | 10.43 | 0.5 | |
| Sodium Carbonate | 3.4 | 29.71 | |
| Sodium | 7.66 | | not tested |
| CO2 | 1.41 | | not tested |
| Inorganic Carbon | 0.38 | | not tested |
| Hydrogen Sulfide | 0.26 | | not tested |
| Total Sulfur as S | 0.84 | 0.41 | |
| Total Carbon | 4.11 | | not tested |
| Water | 81.5 | | not tested |
| Total Hydrocarbon | 0 | | not tested |
| Total Mercaptans | 0.34 | | not tested |
| Cresols | 1.43 | 0.38 | |
| Phenol | 0.67 | 0.44 | |
| Alkyl Phenols | 1.39 | | not tested |

[1] The total of all percentage for the feed sample is greater than one-hundred because some elements are double counted within various compounds.

The results of two subsequent pilot tests were similar. In each test the oil that was recovered after the combined deodorization/evaporation process had concentrated the total volume of feed material was between 10% and 15% of the total volume of feed material processed.

During the evaporation process the carbon dioxide contained in the combustion gas injected into the aqueous spent caustic liquid reacted with the water to produce carbonic acid in accordance with the following equation:

$$CO_2 + H_2O = H_2CO_3 \quad (1)$$

and the carbonic acid reacted with the sodium hydroxide to produce sodium carbonate and water in accordance with the following equation:

$$2NaOH + H_2CO_3 = Na_2CO_3 + 2H_2O \quad (2)$$

From the foregoing it will be seen that each mol of carbon dioxide in the combustion gas injected into the caustic liquid converts two mols of sodium hydroxide to one mol of sodium carbonate and one mol of water. The molecular weight of carbon dioxide is 44 and that of sodium hydroxide is 80, while the molecular weight of carbonic acid is 62, that of sodium carbonate is 106 and that of water is 18. Accordingly, 10,000 lbs. of a 10% sodium hydroxide solution produces 1,325 lbs. of sodium carbonate and 450 lbs. of water and consumes 550 lbs. or 4,488 standard cubic feet of carbon dioxide. Consequently, with a spent caustic feed that weighs 9.237 lbs. per gallon containing 10% of sodium hydroxide (w/w), each 10,000 gallons treated sequesters 1,451 cubic feet of carbon dioxide, or for an average of 30,000 gallons per day of spent caustic effluent treated, 2,781 tons of carbon dioxide per year are sequestered and therefore prevented from release to the atmosphere. Thus, the commercial value of the process includes the direct environmental and economic benefits, i.e., potential revenue for managing spent caustic effluent, the potential sale of recovered product or products, the mitigation of greenhouse gas emissions with the potential for generating commercially valuable "carbon credits" in direct proportion to the quantity of greenhouse gas that is sequestered. If desired, caustic liquid which is a commercially available product rather than a spent caustic effluent may be supplied to the evaporator to sequester carbon dioxide.

The method may be used to process caustic effluents with or without the recovery of products or solely for the purpose of sequestering carbon dioxide contained in the combustion gas supplied to the evaporator.

Although the invention has been described herein with reference to specific embodiments, many modifications and variations therein will readily occur to those skilled in the art. Accordingly, all such variations and modifications are included within the intended the scope of the invention.

WE CLAIM:

1. A method for treating spent caustic effluents comprising:

supplying a spent caustic effluent to a submerged combustion gas evaporator; and

supplying combustion gas to the submerged combustion gas evaporator at a temperature and quantity high enough to vaporize water and volatile constituents in the spent caustic effluent and concentrate a constituent of the spent caustic effluent.

2. A method according to claim 1 including thermally oxidizing vaporized constituents removed from the spent caustic effluent to eliminate odorous and other volatile materials from gaseous constituents released to the atmosphere.

3. A method according to claim 1 wherein the spent caustic effluent includes an aqueous caustic liquid and oil and including the step of separating at least some of the oil from the aqueous constituent in an immiscible liquid separator.

4. A method according to claim 3 wherein the immiscible liquid separator comprises at least one of a gravity settling tank, an inclined plate separator and a centrifuge.

5. A method according to claim 1 wherein the combustion gas supplied to the submerged combustion gas evaporator contains carbon dioxide and including the step of reacting the carbon dioxide with a caustic constituent of the spent caustic effluent to convert the caustic constituent to a carbonate, thereby sequestering carbon dioxide from the combustion gas.

6. A method according to claim 1 wherein the content of caustic material in the caustic waste liquid supplied to the combustion gas evaporator has a sodium hydroxide equivalency within a range from about one percent to about 50% by weight.

7. A method according to claim 1 including maintaining the spent caustic effluent in the submerged combustion gas evaporator at a level between about 5

inches and about 100 inches above the level at which the combustion gas is injected into the evaporator.

8. A method according to claim 1 including maintaining an operating pressure within the submerged combustion gas evaporator in a range from about 50 inches negative to about 100 inches positive of water.

9. A method according to claim 1 including supplying a fuel and air mixture at a pressure in a range from about 5 inches to about 200 inches of water to a burner to produce the combustion gas.

10. A method according to claim 1 including supplying a stack gas from a separate combustion process to the submerged combustion gas evaporator at a pressure in a range from about 60 inches negative to about 100 inches positive of water.

11. A method according to claim 1 including maintaining an operating temperature within the submerged combustion gas evaporator within a range from about 100° F to about 250° F.

12. A method according to claim 1 wherein the spent caustic effluent contains oil and including the step of removing at least some oil from the caustic waste liquid before supplying the caustic waste liquid to the submerged combustion gas evaporator.

13. A method according to claim 1 including supplying a sulfidic spent caustic effluent to the submerged combustion gas evaporator.

14. A method according to claim 1 including supplying a cresylic spent caustic effluent to the submerged combustion gas evaporator.

15. A method according to claim 1 including supplying a napthenic spent caustic effluent to the combustion gas evaporator.

16. A method according to claim 1 including supplying any combination of sulfidic, cresylic and napthenic spent caustic effluents to the combustion gas evaporator.

17. A method according to claim 1 including sequestering at least some carbon dioxide from the combustion gas by converting a caustic effluent constituent from a hydroxide to a carbonate.

18. A method according to claim 1 including stripping at least some volatile compounds from the spent caustic effluent in an air stripper prior to supplying the spent caustic effluent to the evaporator and supplying air from the air stripper containing volatile compounds to a burner used to produce the combustion gas.

19. A method for treating caustic materials comprising supplying a caustic solution which has a sodium hydroxide equivalency in a range from about one percent to about 50 percent to a submerged combustion gas evaporator and supplying combustion gas which contains carbon dioxide to the combustion gas evaporator and wherein the carbon dioxide reacts with the waste liquid to convert a hydroxide constituent thereof to a carbonate, thereby sequestering carbon dioxide from the combustion gas.

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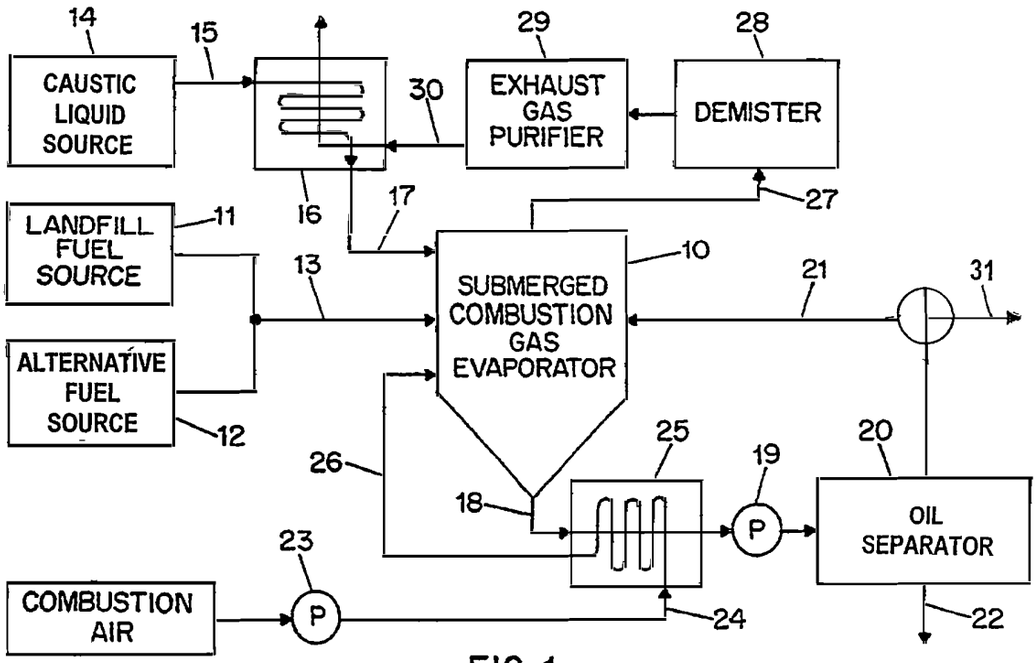


FIG. 1

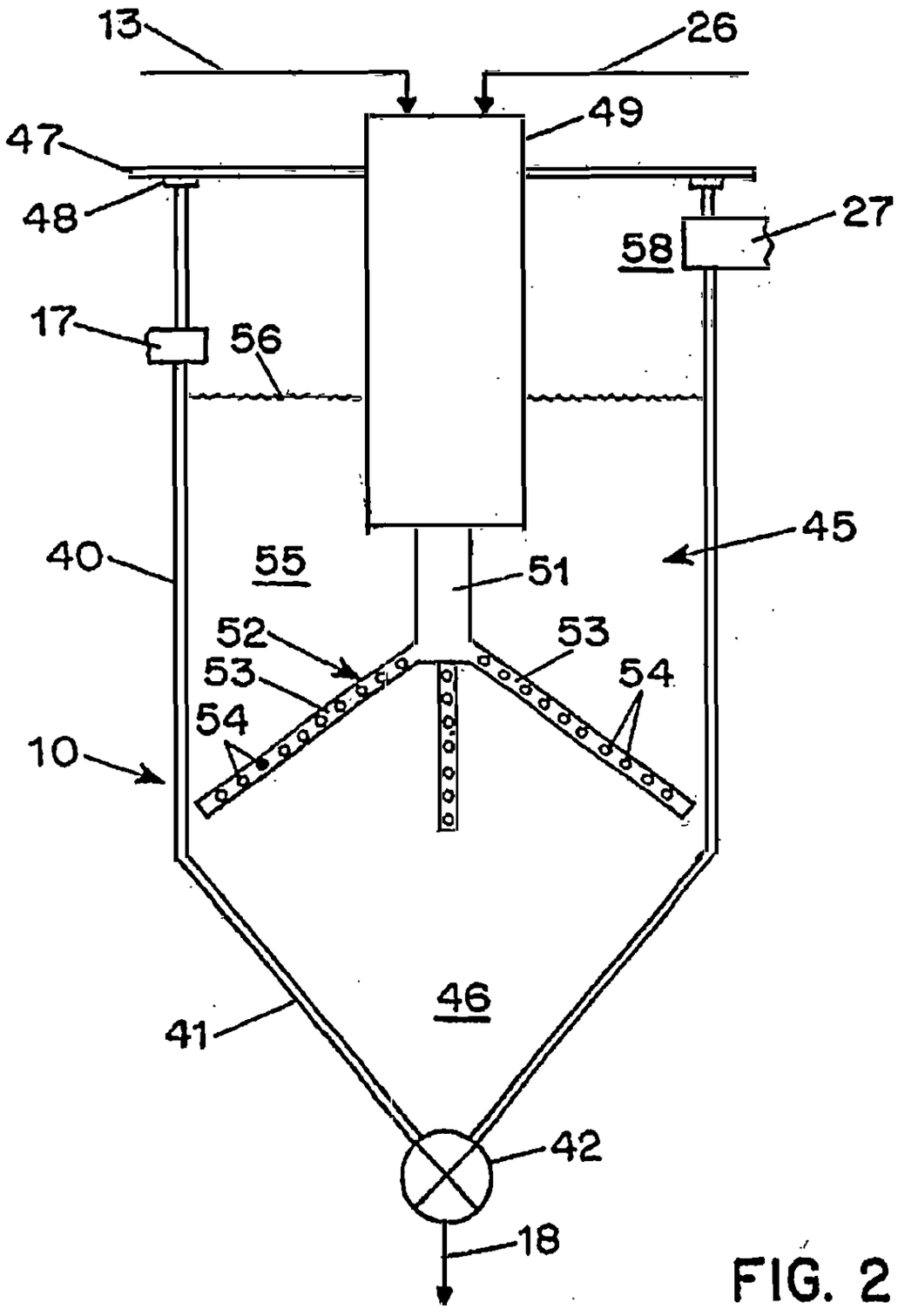


FIG. 2

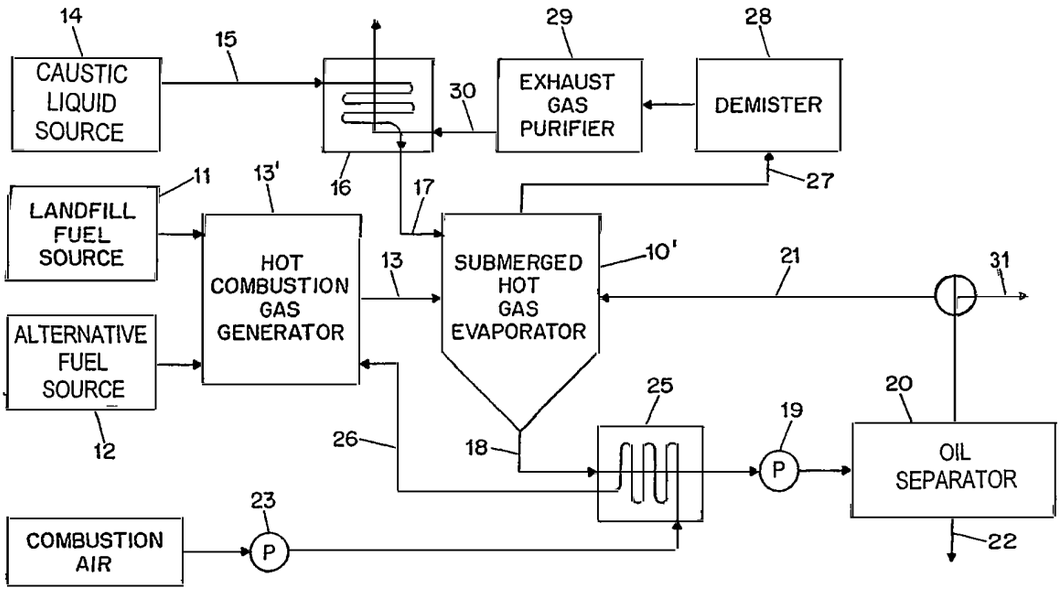
14
CAUSTIC LIQUID SOURCE
15
29
EXHAUST GAS PURIFIER
28
DEMISTER
30
11
LANDFILL FUEL SOURCE
13'
16
17
27
HOT COMBUSTION GAS GENERATOR
13
SUBMERGED HOT GAS EVAPORATOR
10'
21
31
ALTERNATIVE FUEL SOURCE
12
25
19
20
26
18
P
OIL SEPARATOR
23
COMBUSTION AIR
P
22

FIG 3

# TRATAMIENTO DE EFLUENTES CÁUSTICOS DE REFINERÍA CONSUMIDOS

## ESPECIFICACIÓN

## SOLICITUD RELACIONADA

Esta solicitud reivindica la prioridad de la solicitud de Patente estadounidense No. 10/234,559, que fue presentada en septiembre 4, 2002, y se incorpora aquí como referencia.

## ANTECEDENTES DE LA INVENCIÓN

Esta invención se relaciona con procesos para tratar efluentes cáusticos consumidos, por ejemplo, efluentes de refinerías de petróleo, fabricación de aluminio, procesamiento de alimento o similares.

Muchas operaciones industriales generan efluentes cáusticos consumidos diluidos tales como procesos para refinar petróleo. Un efluente cáustico consumido diluido típico de tales refinerías puede contener cerca de 5% a 12% de hidróxido de sodio (p/p) con cantidades que varían

pero significativas de compuestos orgánicos que incluyen un rango de mercaptanos, aceites sulfídicos, ácidos nafténicos, ácidos cresílicos y derivados. Incluidos en la mezcla están menos cantidades de otros compuestos orgánicos e inorgánicos. En adición, un efluente de refinería de petróleo puede contener aproximadamente 5% a 20% por volumen de aceite junto con solución cáustica acuosa. Tradicionalmente tales efluentes han sido considerados esencialmente corrientes de desperdicio inútiles pero han requerido procesamiento significativo antes de desechar para evitar contaminación al medio ambiente.

La Patente otorgada a Helleur No. 4,079,585 describe un método y un aparato para remoción y recuperación de un constituyente a partir de corrientes de desperdicio municipal e industrial mediante evaporación y concentración del constituyente como un resultado de contacto turbulento e íntimo entre el líquido de la corriente de desperdicic y gases calientes producidos por combustión en una torre depuradora y los estados que las técnicas de combustión sumergidas también pueden ser utilizadas como ventaja en el proceso. De acuerdo con tal Patente los polulantes volátiles no deseados pueden ser

removidos sin vaporizarlos mediante la adición de un álcali tal como lima o cáustica para retener los polulantes volátiles acídicos tales como dióxido de azufre, sulfuro de hidrógeno, etc., en solución, de tal forma que ellos pueden ser dispuestos en forma de líquido. La Patente de Helleur describe el proceso como siendo aplicable a material consumido de tales industrias como la industria del aceite y nota que, para evitar la vaporización de volátiles de combustible seleccionados, la temperatura de los gases de combustión se debe mantener por debajo del punto de inflamación de los volátiles mediante el enfriamiento de gas de combustión antes de entrar en contacto con el líquido.

La Patente de Young et al. No. 4,16,028 describe un evaporador de combustión sumergido como la primer etapa en un proceso para la concentración de corrientes de desperdicio industriales.

En la Patente de Ohkawa No. 3,966,594, el tratamiento del agua de desperdicio que contiene sustancias orgánicas solubles en agua en varias formas se describe y el método de combustión sumergido se establece para ser insuficiente industrialmente en términos de concentración

y combustión. En cambio, tal patente describe un proceso en el que el agua de desperdicio se trata con una solución disolvente orgánico insoluble en agua de un constituyente orgánico.

De acuerdo con la Patente de Anderson No. 4,188,291 el agua de desperdicio industrial es procesada mediante un evaporador de combustión sumergido y dióxido de carbono en los gases de combustión suministrado al agua de desperdicio es secuestrado por hidróxido de calcio que ha sido agregado para producir carbonato de calcio que es luego separado de la corriente de desperdicio.

El proceso de tratamiento cáustico consumido descrito en la Patente DeRoeck et al. No. 5,244,576 Introduce gases de refinería que contienen dióxido de carbono y sulfuro de hidrógeno dentro de una solución de hidróxido de sodio para convertir el dióxido de carbono a carbonato de sodio.

En la patente de Connally No. 3,985,609 la concentración de constituyentes en un líquido a ser concentrado se efectúa al suministrar el líquido a un evaporador de combustión sumergido.

La Patente de Echols No. 5,934,207 describe la evaporación de lixiviados al dirigir llamas de un quemador al que se suministra gas de relleno dentro de un calentador de tubo de fuego inmerso en un tanque que contiene lixiviados de tal forma que calienta y vaporiza los constituyentes líquidos en el lixiviado mientras se dispone del gas de relleno.

La Patente de Duesel No. 5,342,482 describe el uso de gas de terraplén como una fuente de combustible para un evaporador de gas de combustión sumergido en un proceso de evaporación de lixiviado.

## RESUMEN DE LA INVENCIÓN

De acuerdo con esto, es un objeto de la presente invención suministrar un proceso para tratar efluentes cáusticos de refinería consumidos que superan las desventajas de la técnica anterior.

Otro objeto de la invención es proporcionar un proceso para tratar elementos cáusticos de una refinería para

convertir un constituyente cáustico a un producto comercial útil.

Un objeto adicional de la invención es suministrar un proceso para tratar efluentes de refinería de petróleo para producir un producto de aceite reutilizable así como también un producto cáustico convertido.

Un objeto adicional de la invención es suministrar un proceso para tratar efluentes cáusticos que utilizan dióxido de carbono en un gas de combustión suministrado al efluente para convertir un constituyente cáustico a un carbonato.

Estos y otros objetos de la invención se logran al suministrar un efluente cáustico a un evaporador de gas de combustión sumergido al que se suministraron gases de combustión en una temperatura y una cantidad seleccionada para remover el vapor no deseado y los gases mientras se retiene un constituyente líquido deseado del efluente, y separar el constituyente deseado de los constituyentes remanentes del efluente. En una modalidad particular de la invención el efluente cáustico es un efluente de refinería de petróleo y el dióxido de carbono en el gas

de combustión suministrado al efluente convierte un constituyente cáustico en el efluente a un carbonato tal como carbonato de sodio. En adición, el aceite se separa de los otros constituyentes efluentes, antes, durante o después de evaporación, para reutilizar o refinar adicionalmente.

De acuerdo con otro aspecto de la invención, al menos algo del combustible utilizado para suministrar el gas de combustión al evaporador de gas de combustión sumergido es gas de relleno y el contenido de dióxido de carbono del gas de combustión se utiliza para convertir el constituyente cáustico del efluente a un carbonato, evitando por lo tanto la diseminación del dióxido de carbono en la atmósfera.

En una modalidad representativa de la invención el efluente consumido suministrado al evaporador de gas de combustión sumergido tiene un contenido de hidróxido de sodio equivalente de cerca de 5% a 12% en peso y un contenido de aceite de cerca de 5% a 20% por volumen y el contenido acuoso del efluente consumido se reduce mediante evaporación como se requiere para producir una concentración de carbonato deseada. El líquido

concentrado puede contener cerca de 20% a 30% de carbonato de sodio y el constituyente de aceite se separa para reutilizar después de concentración mediante un separador por gravedad.

## BREVE DESCRIPCIÓN DE LOS DIBUJOS

Objetos y ventajas adicionales de la invención serán evidentes a partir de la lectura de la siguiente descripción en conjunto con los dibujos que la acompañan en los cuales:

La figura 1 es un diagrama de bloques esquemáticos que ilustra una modalidad representativa de una disposición para llevar a cabo un proceso para tratar efluentes cáusticos de acuerdo con la invención;

La figura 2 es una vista seccional esquemática que ilustra una modalidad representativa de un evaporador de gas de combustión sumergido para uso en llevara cabo el proceso de la invención; y

La figura 3 es un diagrama de bloques esquemáticos que ilustra una modalidad representativa adicional de una

disposición para tratar efluentes cáusticos de acuerdo con la invención.

## DESCRIPCIÓN DE LAS MODALIDADES PREFERIDAS

En una disposición representativa para llevar a cabo el proceso de la invención ilustrada esquemáticamente en la figura 1, un evaporador de gas de combustión sumergido 10, que se describe en más detalle en relación con la figura 2, recibe el gas del combustible de un relleno u otra fuente de biogás 11 o de una fuente de combustible alternativa 12 que proviene de gas natural, propano, butano o similar o que suministra un combustible líquido tal como aceites de petróleo de desperdicio o similares o de ambos. El evaporador 10 puede ser operado sobre una tanda, tandas continuas, o sobre una base continua y el gas de combustible de las fuentes 11 y 12 puede ser recolectado y almacenado para uso o ser suministrado continuamente de acuerdo con las necesidades del evaporador. En una modalidad del sistema se ubica cercanamente adyacente a un relleno del cual los gases del relleno están disponibles convenientemente pero, si se desea, los gases del relleno pueden ser enviados por tubería o transportados de uno o más rellenos en

ubicaciones remotas a estaciones de procesamiento adyacentes a una fuente de líquido efluente cáustico tal como una refinería u otras instalaciones de fabricación.

En la modalidad ilustrada en la figura 1 un líquido cáustico de una fuente 14 tal como un efluente cáustico consumido de una refinería de petróleo, una planta de fabricación de aluminio o instalaciones de procesamiento de alimentos se transmite a través de una línea 15 a una unidad de intercambiador de calor 16 para precalentar mediante gases de exhosto caliente y luego enviadas por tubería a través de una línea 17 a la entrada al evaporador de gas de combustión sumergido 10. El cáustico en el efluente consumido puede ser hidróxido de sodio o cualquier otro metal alcalino y el efluente consumido puede ser de una refinería de petróleo, una planta de fabricación de aluminio, instalaciones de procesamiento de alimentos o de varias operaciones industriales tales como depuración. La equivalencia de hidróxido de sodio del material cáustico en el efluente consumido de la fuente 14 está preferiblemente en un rango de cerca de 1% a cerca de 50%, deseablemente en el rango de cerca de 1% a 15% y más deseablemente en un rango de cerca de 2% a 12%. Si el efluente consumido es de una refinería de

petróleo y contiene una proporción sustancial de aceite, el efluente consumido puede ser pasado a través de un aceite en forma preliminar y separador de agua (no mostrado) para remover al menos una porción del aceite antes que sea administrado al evaporador 10.

Luego de la evaporación de los constituyentes vaporizables del efluente consumido en el evaporador 10, el líquido concentrado es removido a través de una salida 18 y transmitido mediante una bomba 19 a través de un separador de aceite 20 para remover el aceite de los constituyentes acuosos del líquido concentrado que pasa del separador a una salida 31. Si es necesario, algunos de todos los constituyentes acuosos pueden ser reciclados a través de una línea 21 de regreso al evaporador 10 para procesar adicionalmente después de la separación del aceite. El aceite separado es removido del separador a través de una línea 22 para venta, además de adicionalmente procesar o reciclar. El separador de aceite 20 puede ser cualquier separador de líquido inmiscible convencional tales como un tanque de sedimentación por gravedad, un separador de placa inclinado o una centrífuga.

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El aire de combustión dirigido por un ventilador 23 y suministrado a través de una línea 24 es precalentado mediante circulación a través de una unidad de intercambio de calor 25 a través del cual el líquido concentrado pasa de la salida 18 y el aire de combustión precalentado es suministrado al evaporador 10 a través de una línea 26. El intercambio de calor en la unidad 25 puede ser utilizado para mejorar la eficiencia del quemador. Si se desea algo de aire de combustión del ventilador 23 puede ser desviado a un separador de aire en línea (no mostrado) en la línea 17 para remover los compuestos volátiles del efluente consumido que está siendo suministrado al evaporador y suministrarlos con el aire de combustión en el evaporador 10. Los gases de exhosto son retirados del evaporador 10 a través de una línea 27 y luego pasados a través de un desempañador 28 en el que los constituyentes de líquidos entrantes son removidos, después que el gas es pasado a través de una unidad de purificación 29 tal como una llama incluida. En la unidad de purificación 29 los compuestos orgánicos volátiles y otros constituyentes de gas de exhosto son térmicamente oxidados o tratados de otra manera para darles menos olor y hacerlos inofensivos antes de ser

liberados en la atmósfera a través de la línea 30 que pasa a través de una unidad de intercambio de calor 16.

La disposición de una forma preferida de evaporador de gas de combustión sumergida para uso en el proceso de la invención se ilustra en la figura 2. Como se muestra en esa figura el evaporador 10 tiene una carcasa 40 con un fondo cónico que converge hacia abajo 41 que conduce a una válvula de descarta 42 a través de la cual el líquido cáustico acuoso concentrado y el aceite, si está presente, pasa dentro de la línea de descarga 18. Dentro de la carcasa 40 los líquidos y sólidos arrastrados, si existen, pueden seguir hacia abajo y ser dirigidos por la porción de carcasa cónica 41 hacia la válvula de descargue 42. En la parte superior de la carcasa 40 una cubierta 47 es sellada con un sello de junta 48 a la pared de la carcasa y un quemador 49 se monta sobre la cubierta.

Los gases de combustión del quemador 49 están dirigidos hacia abajo a través de una tubería descendente de gas de combustión 51 dentro de un distribuidor 52 que tiene una serie de tubos de aspersión de mezcla 53. Los tubos del distribuidor tienen el orificio 54 a través de los cuales

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los gases de combustión calientes son inyectados dentro de un tanque 55 del efluente cáustico consumido contenido dentro de la carcasa de tal forma que evapora el líquido de este en una zona de evaporación 45 mediante transferencia de calor de contacto directo, suministrando y distribuyendo dióxido de carbono al efluente consumido para efectuar la conversión a carbonato y también elimina los constituyentes vaporizables. Preferiblemente, la presión de operación dentro del evaporador está en un rango de cerca de 50 pulgadas negativa a 100 pulgadas positivas de agua.

La superficie superior 56 del tanque de líquido de desperdicio cáustico dentro de la carcasa es mantenido en un nivel deseado al controlar el suministro de efluente consumido admitido dentro del evaporador a través de la línea 17 de la fuente 14. preferiblemente, el nivel de la superficie superior 56 está en un rango de cerca de 5 pulgadas a cerca de 100 pulgadas sobre el distribuidor 52 y la temperatura del tanque de efluente consumido 55 está en un rango de cerca de 100° F a cerca de 250° F. Si el evaporador está siendo operado sobre una base continua el efluente consumido es suministrado al evaporador continuamente en un índice apropiado de tal forma que

mantiene la superficie 56 en un nivel indicado dentro del evaporador.

De otra parte, si el evaporador está siendo operado sobre una base de tanda el efluente a ser procesado será suministrado continuamente en un índice apropiado para mantener la superficie 56 en un nivel indicado hasta que la concentración de constituyentes deseados en el efluente alcanza un valor seleccionado. Después de eso se termina el suministro y el evaporador es ya sea apagado o vaciado o la operación puede continuar hasta que la superficie del efluente dentro del evaporador cae a un nivel inferior (no mostrado) que está sobre la parte más alta del distribuidor de gas 52 en cuyo tiempo el evaporador se apagará y el efluente concentrado se descargará, después de lo cual el evaporador puede ser encendido de nuevo.

En una modalidad preferida la temperatura de entrada de los gases de combustión en el distribuidor 52 están en un rango de cerca de 600° F a 1.800° F, la temperatura y cantidad son suficientemente altas para vaporizar el agua y los constituyentes volátiles en el efluente consumido para concentrar los constituyentes concentrados deseados

tales como el aceite y/o un cáustico convertido en una proporción deseada.

Cuando se opera en una forma de tanda continua la operación procede como en un modo continuo excepto que los efluentes concentrados son removidos periódicamente de la descarga 42 sin reducir el nivel de líquido o apagar el evaporador. Como se indica en la figura 1, el combustible es suministrado al quemador 49 a través de una línea 13 y el aire de combustión es suministrado al quemador a través de la línea 26 bajo presión suficiente, preferiblemente en un rango de cerca de 5 pulgadas a cerca de 200 pulgadas de agua, para proveer combustión eficiente y forzar a los gases de combustión a través del distribuidor 52 y transportar los gases de exhosto a través de la línea de exhosto 27.

Se debe notar que, exclusivo de las bombas de proceso, solo un miembro de movimiento sencillo se requiere para llevar a cabo este proceso dado que un suministro de combustible está disponible en la presión adecuada, es decir, el ventilador 23 que suministra aire de combustión a presión reubica. De acuerdo con esto, el proceso de tratamiento de efluente cáustico consumido de la

invención no solo remueve constituyentes olorosos del efluente sino que también produce un concentrado acuoso útil comercialmente y separa un constituyente de aceite acuoso y una forma simple y efectiva sin las partes de movimiento complejo que se requieren que conducen a modalidades difíciles en la técnica anterior de sistemas que manejan efluentes cáusticos consumidos.

Más aún, aunque el efluente cáustico consumido es pasado a través de las unidades de intercambio de calor 16 y 25 en la disposición descrita en la figura 1, las dificultades que resultan del uso de la técnica anterior de evaporadores de intercambio de calor no se encuentran en aquellas unidades ya que no se lleva a cabo la evaporación en ellas y se evita el consecuente ensuciamiento de las superficies del intercambiador de calor. En formas simples de sistemas de tratamiento de efluente cáustico consumido de acuerdo con la invención, las unidades de recuperación de calor 16 y 25 pueden ser omitidas, si el vapor de exhosto presenta estándares ambientales para descarga directa a la atmósfera, las unidades de tratamiento de exhosto 28 y 29 también pueden ser omitidas.

La figura 3 ilustra una modalidad alternativa que es idéntica a aquella de la figura 1 excepto que, como se describió previamente, una fuente de gas de calor espaciados del evaporador tales como un generador de gas de calor 13' que puede por ejemplo ser un motor de combustión interna, suministra gases calientes a través de la línea 13 al dispositivo de distribución de gas caliente 52 en un evaporador de gas caliente sumergido modificado 10', la línea de suministro de aire 26 es conectada al generador de gas caliente 13' en forma diferente al evaporador 10'. En este caso los gases calientes del generador de gas caliente son suministrados al evaporador en una presión en el rango en el rango de 60 pulgadas negativas a cerca de 120 pulgadas positivas de agua. En todos los otros aspectos el sistema de tratamiento líquido cáustico de la figura 13 es el mismo como aquel mostrado en la figura 1 y el evaporador 10' es el mismo como el evaporador 10 ilustrado en la figura 2.

De acuerdo con la invención, el efluente cáustico consumido de refinería de petróleo o similares puede ser concentrado eficientemente y efectivamente sin requerir un evaporador de intercambio de calor del tipo utilizado en los sistemas de tratamiento de efluente cáustico

consumido convencionales que tienen superficies que pueden ser ensuciadas por los residuos de efluente y por lo tanto requerir limpieza periódica o reemplazo. Los efluentes cáusticos consumidos de refinería de petróleo que pueden ser procesados mediante la presente invención incluyen efluentes sulfídicos, cresílicos y nafténicos.

Como se describió en el documento titulado "Effluent Caustic Treating Systems Using MERICON$^{SM}$ Technologies" de Merichem Chemicals and Refinery Services LLC., los efluentes cáusticos sulfídicos son producidos por gas de combustible, gas de petróleo licuificado (LPG) y procesos de tratamiento de gasolina. Una corriente cáustica sulfídica de refinería típica tiene la composición mostrada en la tabla 1 adelante. Los contaminantes usuales son sulfuro de sodio y mercapturo de sodio. Estos compuestos originan alta demanda de oxígeno químico y biológico en los procesos de tratamiento y producen olores y gases dañinos cuando no se neutralizan.

Tabla 1

| | |
|---|---|
| NaOH libre, % en peso | 2 a 10 |
| Sulfuros y bisulfuros como S, % en peso | 0,5 a 4 |
| Mercapturos como S, % en peso | 0,1 a 4 |
| Carbonatos como $CO_3$, % en peso | 0 a 4 |
| pH | 13 a 14 |
| Amonio | Traza |

Los usos comerciales de las soluciones cáusticas sulfídicas concentradas de acuerdo con la invención son como agentes de tratamiento en procesamiento de pulpa y papel y para purificar ciertos minerales de metal en la industria minera.

Los efluentes cáusticos cresílicos, que pueden contener aceites de ácido aromático, son producidos al tratar cáusticos de gasolina craqueada y destilados craqueados. Las corrientes cáusticas cresílicas contienen fenoles, cresoles, y otros ácidos orgánicos que están generalmente presentes en cresilatos solubles en agua que se separan de los cáusticos como aceites ácidos en un pH neutral. Las soluciones cáusticas cresílicas producidas de tratar

gasolinas craqueadas generalmente viene de dos fuentes: 1) extracción de mercaptanos o sistemas de oxidación que utilizan cáusticos fuertes; y 2) sistemas de oxidación de mercaptano que utilizan cáusticos débiles. La tabla 2 adelante muestra las características de efluentes cáusticos cresílicos típicos.

Tabla 2

| | Operación cáustica fuerte | Operación cáustica diluida |
|---|---|---|
| NaOH, % en peso | 10 a 15 | 1 a 4 |
| Sulfuros como S. % en peso | 0 a 1 | 0 a 0,2 |
| Mercapturos como S, % en peso | 0 a 4 | 0 a 0,5 |
| Ácidos cresílicos, % en peso | 10 a 25 | 2 a 5 |
| Carbonatos como $CO_3$, % en peso | 0 a 0,5 | 0 a 0,1 |
| pH | 12 a 14 | 12 a 14 |

Las soluciones cáusticas cresílicas concentradas tienen valor comercial como intermedios químicos.

Las soluciones cáusticas nafténicas son generadas de tratamiento cáustico de cortes de kerosén y diesel de una pizarra cruda nafténica. La tabla 3 adelante muestra características típicas de corriente de naftenato derivadas de depósitos de kerosén y diesel.

Tabla 3

| | Combustible Jet/kerosén | Diesel |
|---|---|---|
| NaOH, % en peso | 1 a 4 | 1 a 4 |
| Sulfuros como S-, % en peso | 0 a 0,1 | Traza |
| Mercapturos como S+, % en peso | 0 a 0,5 | 0 a 0,5 |
| Ácidos Nafténicos, % en peso | 2 a 10 | 2 a 15 |
| Cresílicos, % en peso | 1 a 3 | 0 a 1 |
| pH | 12 a 14 | 12 a 14 |

Las soluciones cáusticas nafténicas concentradas tienen valor comercial potencial para procesadores quienes los refinan para venta para productores de sales de metal de naftenato.

Las refinerías de petróleo típicamente pagan costos por embarque, tratamiento y depósito de líquidos de desperdicio industrial de producción que incluyen cáusticos consumidos. Las refinerías podrían encontrar útil rescatar ambas el líquido cáustico y el aceite para reutilizarlo en su proceso si ellos pueden ser desodorizados y la equivalencia de hidróxido de sodio concentrada para valores aceptables. De acuerdo con la presente invención, el cáustico consumido de refinería de petróleo puede ser procesado para satisfacer aquellos requerimientos efectiva y eficientemente.

En un ensayo piloto del proceso de la invención, se obtuvo una reducción significativa en el nivel de olor en el líquido concentrado comparado con el material de alimentación. El líquido concentrado fue una mezcla de dos fases de aceite y una fase acuosa que puede ser

fácilmente separada mediante separación por gravedad tal como decantación. Después de decantar, en ensayo de fase acuosa mostró que una concentración significativa de cáustico se alcanzó. El ensayo también mostró casi conversión del 100% de hidróxido de sodio en el efluente cáustico consumido del carbonato de sodio debido a la reacción del dióxido de carbono en el gas de combustión con el hidróxido como se discutió aquí anteriormente. En este ensayo la concentración de carbonato de sodio en la fase acuosa concentrada fue aproximadamente 30% (p/p) a este respecto, la aspersión de gas combustible directa que tiene lugar en el evaporador de gas de combustión sumergido durante el proceso origina la formación de ácido carbónico en el agua que está presente dentro del evaporador durante el procesamiento y el ácido carbónico entonces reacciona con el constituyente cáustico, hidróxido de sodio, para formar carbonato de sodio en la forma descrita adelante.

El material de alimentación cáustico diluido para el ensayo piloto fue una mezcla aproximadamente 1:1 de dos productos de operaciones de refinería conocidos como corrientes cáusticas gastadas "sulfídica" y "cresílica". Los ensayos de cada una de estas corrientes y de los

materiales utilizados como alimentación para el ensayo piloto se presentan en la tabla 4 adelante.

Tabla 4

Composición de Corrientes de Alimentación Constituyente y Mezcla Promedio

| CÁUSTICO DILUIDO | Corriente 1 | Corriente 2 | Alimentación de Unidad Piloto |
|---|---|---|---|
| | Sulfuro | Cresilato | (Mezcla de Corrientes 1 & 2 1:1) |
| Resultados de ensayo, %[1] | | | |
| Hidróxido de sodio | | | |
| Carbonato de sodio | | | |
| Sodio | | | |
| CO2 | | | |
| Carbón inorgánico | | | |
| Sulfuro de Hidrógeno | | | |

| | | | |
|---|---|---|---|
| Total de Azufre como S | | | |
| Total Carbono | | | |
| Agua | | | |
| Total hidrocarburo | | | |
| Total mercaptanos | | | |
| Cresoles | | | |
| Fenol | | | |
| Fenoles álcali | | | |

[1] El total de los porcentajes en mayor que cien ya que algunos elementos se cuentan dos veces dentro de varios compuestos.

La meta del ensayo piloto fue desodorizar y concentrar el contenido de hidróxido de sodio de un material cáustico que fue producido como un producto en operaciones de refinería de petróleo. Ya que el gas de combustión suministrado al evaporador contenía dióxido de carbono, este fue anticipado de alguna porción del hidróxido de sodio que sería convertido a carbonato de sodio. No se

conoció como la mezcla de aceites llevados dentro del material de alimentación cáustica pudo afectar, o ser afectado por, el proceso.

Los resultados del ensayo piloto mostraron que el hidróxido de sodio ha sido convertido casi completamente a carbonato de sodio, y que una fase de aceite fue producida que fue inmiscible con la fase de carbonato de sodio acuoso. Decantar fácilmente separadas las dos fases inmiscibles. Cada una de las fases producidas en el experimento fue juzgada por tener niveles reducidos significativamente de olor comparado con el material de alimentación, demostrando que el objetivo de desodorizar la alimentación cáustica diluida durante el proceso se ha alcanzado.

La tabla 5 adelante muestra una comparación de constituyentes seleccionados en el material utilizado como alimentación de la unidad piloto y la fase acuosa produjo como un resultado de la sujeción del material al proceso de desodorización/evaporación combinada. En el ensayo, el volumen del material de alimentación fue reducido a aproximadamente una tercera parte del volumen de líquido efluente cáustico consumido original.

Tabla 5

Comparación de Material de Alimentación con Concentrado

| Fase Acuosa del Primer Ensayo Piloto | Alimentación | Final | Comentarios |
|---|---|---|---|
| | | | |
| Resultados del Ensayo, %[1] | | | |
| | | | |
| Hidróxido de Sodio | 10,43 | 0,5 | |
| Carbonato de Sodio | 3,4 | 29,71 | |
| Sodio | 7,66 | | No ensayado |
| CO2 | 1,41 | | No ensayado |
| Carbón Inorgánico | 0,38 | | No ensayado |
| Sulfuro de Hidrógeno | 0,26 | | No ensayado |
| Total Azufre como S | 0,84 | 0,41 | |
| Total Carbono | 4,11 | | No ensayado |
| Agua | 81,5 | | No ensayado |
| Total Hidrocarburo | 0 | | No ensayado |
| Total Mercaptanos | 0,34 | | No ensayado |
| Cresoles | 1,43 | 0,38 | |

| | | | |
|---|---|---|---|
| Fenol | 0,67 | 0,44 | |
| Fenoles alquilo | 1,39 | | No ensayado |

[1] El total de todos los porcentajes para la muestra de alimentación es mayor de cien debido a que algunos de los elementos se contaron dos veces dentro de varios compuestos.

Los resultados de dos ensayos piloto posteriores fuero similares. En cada ensayo el aceite que fue recuperado después del proceso de desodorización/combinado ha concentrado el volumen total de material de alimentación que estuvo entre 10% y 15% del total de volumen de material de alimentación procesado.

Durante el proceso de evaporación el dióxido de carbono contenido en el gas de combustión inyectado dentro del líquido cáustico consumido acuoso se hizo reaccionar con el agua para producir ácido carbónico de acuerdo con la siguiente ecuación:

$$CO_2 + H_2O = H_2CO_3 \qquad (1)$$

Y el ácido carbónico se hizo reaccionar con el hidróxido de sodio para producir carbonato de sodio y agua de acuerdo con la siguiente ecuación:

$$2NaOH + H_2CO_3 = Na_2CO_3 + 2H_2O \qquad (2)$$

A partir de lo anterior se verá que cada mol de dióxido de carbono en el gas de combustión inyectado dentro el líquido cáustico convierte dos moles de hidróxido de sodio a un mol de carbonato de sodio y 1 mol de agua. El peso molecular de dióxido de carbono es 44 y de hidróxido de sodio es 80, mientras el peso molecular del ácido carbónico es 62, de carbonato de sodio es 106 y de agua es 18. De acuerdo con esto, 10.000 lbs de un 10% de solución de hidróxido de sodio produce 1.325 lbs de carbonato de sodio y 450 lbs de agua y consume 550 lbs o 4.488 pies cúbicos estándar de dióxido de carbono. Posteriormente, con una alimentación cáustica gastada que pesa 9,237 lbs por galón que contiene 10% de hidróxido de sodio (p/p) cada 10.000 galones de secuestrante tratado 1.451 pies cúbicos de dióxido de carbono, o para un promedio de 30.000 galones por día de un efluente cáustico consumido tratado, 2.781 toneladas de dióxido de carbono por año son secuestradas y por lo tanto se

evitada liberarse a la atmósfera. Así, el valor comercial del proceso incluye beneficios ambientales y económicos directos, es decir, ingreso potencial para manejar efluente cáustico consumido, la venta potencial de productos recuperados o productos, la mitigación de emisiones de gas de invernadero con el potencial de generar comercialmente "créditos de carbono" comercialmente valiosos en proporción directa a la cantidad de gas de invernadero que es secuestrado. Si se desea, el líquido cáustico que es un producto disponible comercialmente diferente del efluente cáustico consumido puede ser suministrado al evaporador al secuestrador dióxido de carbono.

El método puede ser usado para procesar efluentes cáusticos con o sin la recuperación de productos o únicamente para proposito de secuestrar dióxido de carbono contenido en el gas de combustión suministrado al evaporador.

Aunque la invención ha sido descrita aquí con referencia a las modalidades específicas, muchas modificaciones y variaciones serán fácilmente hechas por aquellos expertos en la técnica. De acuerdo con esto, todas las variaciones

y modificaciones se incluyen dentro del alcance propuesto de la invención.

## REIVINDICACIONES

1. Un método para tratar efluentes cáusticos consumidos que comprende:

suministrar un efluente cáustico consumido a un evaporador de gas de combustión sumergido; y

suministrar gas de combustión al evaporador de gas de combustión sumergido en una temperatura y cantidad suficientemente alta para evaporar agua y constituyentes volátiles en el efluente cáustico consumido y concentrar un constituyente del efluente cáustico consumido.

2. Un método de acuerdo con la reivindicación 1 que incluye oxidar térmicamente constituyentes vaporizados removidos del efluente cáustico consumido para eliminar olores y otros materiales volátiles de constituyentes gaseosos liberados a la atmósfera.

3. Un método de acuerdo con la reivindicación 1 en donde el efluente cáustico consumido incluye un

líquido cáustico acuoso y aceite e incluye la etapa de separar al menos de alguno del aceite del constituyente acuoso en un separador de líquido inmiscible.

4. Un método de acuerdo con la reivindicación 3 en donde el separador de líquido inmiscible comprende al menos uno de un tanque de sedimentación por gravedad, un separador de placa inclinada y una centrífuga.

5. Un método de acuerdo con la reivindicación 1 en donde el gas de combustión suministrado al evaporador de gas sumergido contiene dióxido de carbono e incluye la etapa de hacer reaccionar el dióxido de carbono con un constituyente cáustico del efluente cáustico consumido para convertir el constituyente cáustico a un carbonato, por lo tanto secuestrando el dióxido de carbono del gas de combustión.

6. Un método de acuerdo con la reivindicación 1 en donde el contenido de material cáustico en el líquido de desperdicio cáustico suministrado al

evaporador de gas de combustión tiene una equivalencia de hidróxido de sodio dentro de un rango de cerca de 1% a cerca de 50% por peso.

7. Un método de acuerdo con la reivindicación 1 que incluye mantener el efluente cáustico consumido en un evaporador de gas de combustión sumergido en un nivel de entre cerca de 5 pulgadas y cerca de 100 pulgadas sobre el nivel en el que el gas de combustión se inyecta dentro del evaporador.

8. Un método de acuerdo con la reivindicación 1 que incluye mantener una presión de operación dentro del evaporador de gas de combustión sumergido en un rango de 50 pulgadas negativas a cerca de 100 pulgadas positivas de agua.

9. Un método de acuerdo con la reivindicación 1 que incluye suministrar una mezcla de combustible y aire en una presión en un rango de cerca de 5 pulgadas a cerca de 200 pulgadas de agua a un quemador para producir gas de combustión.

10. Un método de acuerdo con la reivindicación 1 que incluye suministrar un gas de un proceso de combustión separador al evaporador de gas de combustión sumergido en una presión en un rango de cerca de 60 pulgadas negativas a cerca de 100 pulgadas positivas de agua.

11. Un método de acuerdo con la reivindicación 1 que incluye mantener una temperatura de operación dentro de un evaporador de gas de combustión sumergido dentro de un rango de cerca de 100° F a cerca de 200° F.

12. Un método de acuerdo con la reivindicación 1 en donde el efluente cáustico consumido que contiene aceite e incluye la etapa de remover al menos algún aceite del líquido de desperdicio cáustico antes de suministrar el líquido de desperdicio cáustico al evaporador de gas de combustión sumergido.

13. Un método de acuerdo con la reivindicación 1 que incluye suministrar un efluente cáustico

consumido sulfídico al evaporador de gas de combustión sumergido.

14. Un método de acuerdo con la reivindicación 1 que incluye suministrar un efluente cáustico consumido cresílico al evaporador de gas de combustión sumergido.

15. Un método de acuerdo con la reivindicación 1 que incluye suministrar un efluente cáustico consumido nafténico al evaporador de gas de combustión.

16. Un método de acuerdo con la reivindicación 1 que incluye suministrar cualquier combinación de efluentes cáusticos consumidos nafténicos cresílicos y sulfídicos al evaporador de gas de combustión.

17. Un método de acuerdo con la reivindicación 1 que incluye secuestrar al menos algún dióxido de carbono del gas de combustión mediante convertir un constituyente efluente cáustico de un hidróxido a un carbonato.

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18. Un método de acuerdo con la reivindicación 1 que incluye separar al menos algunos compuestos volátiles del efluente cáustico consumido en un separador de aire antes de suministrar el efluente cáustico consumido al evaporador y suministrar aire del separador d aire que contiene compuestos volátiles a un quemador utilizado para producir gas de combustión.

19. Un método para tratar materiales cáusticos que comprende suministrar una solución cáustica que tiene una equivalencia de hidróxido de sodio en un rango de cerca de 1% a cerca de 50% a un evaporador de gas de combustión sumergido y suministrar gas de combustión que contiene dióxido de carbono al evaporador de gas de combustión y en donde el dióxido de carbono reacciona con el líquido de desperdicio para convertir un constituyente de hidróxido de este a un carbonato, secuestrando por lo tanto el dióxido de carbono del gas de combustión.

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**RESUMEN**

En los métodos para tratamiento de efluentes cáusticos descritos en la especificación, un efluente de refinería cáustico consumido se suministra a un evaporador de gas de combustión sumergido en el que el gas de combustión caliente que contiene dióxido de carbono se inyecta en el líquido cáustico para concentrar el líquido y convierte un constituyente hidróxido a un carbonato. En donde el efluente cáustico es de una refinería de petróleo, el aceite en el líquido de desperdicio es separado del constituyente acuoso antes, durante y después de concentración.

WO 2004/022194 PCT/US2003/027517



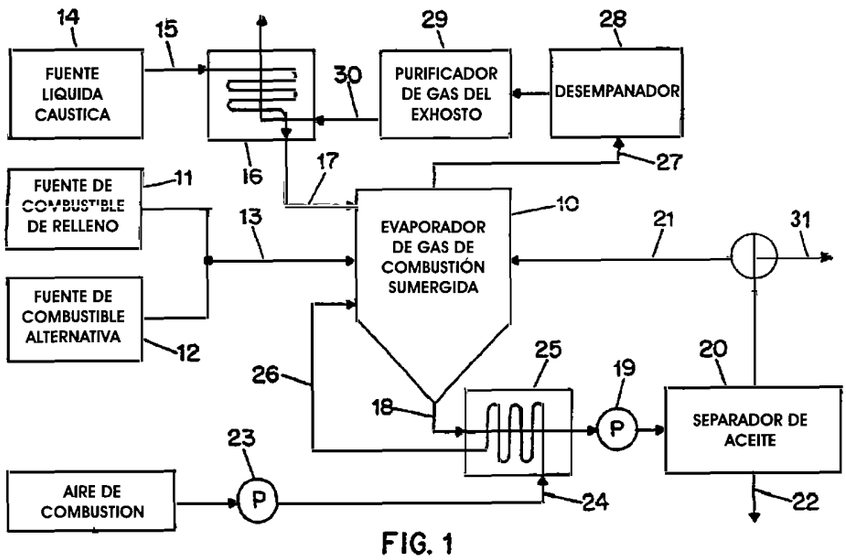


FIG. 1

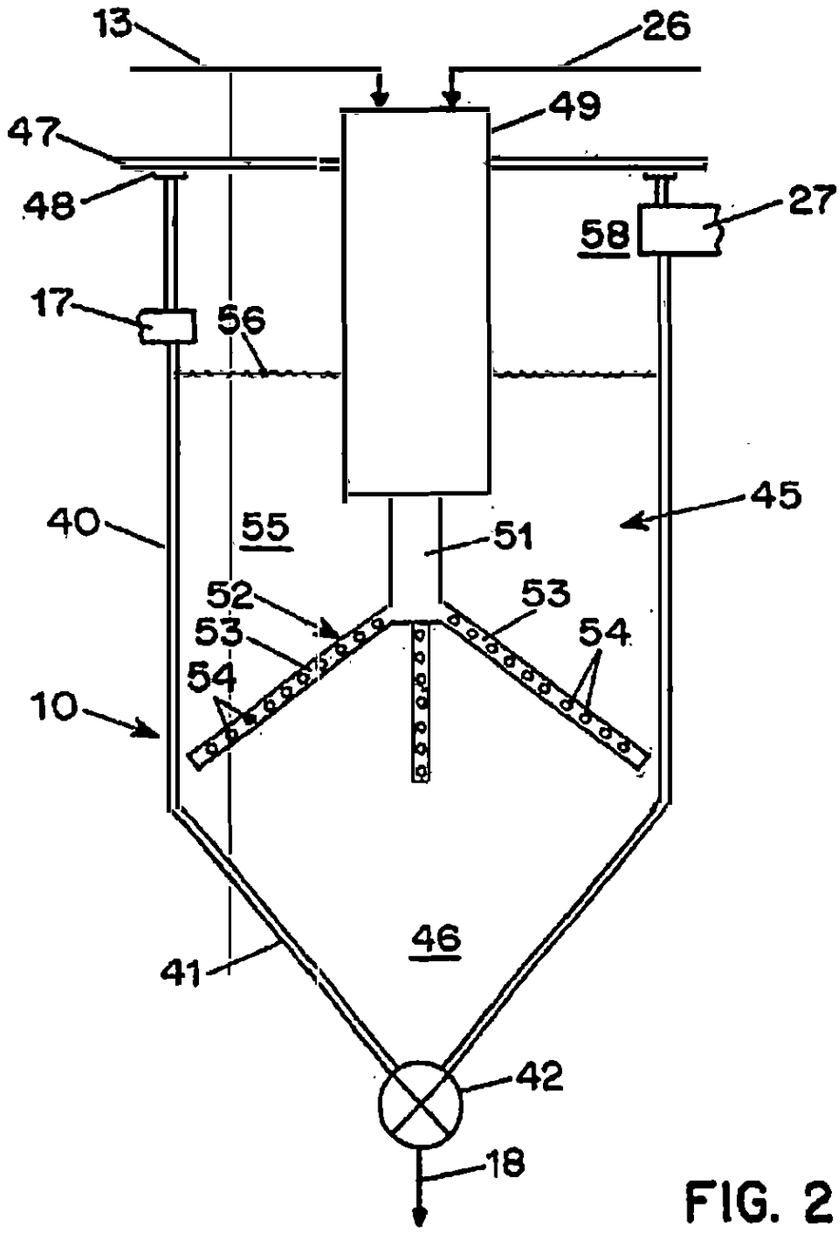


FIG. 2

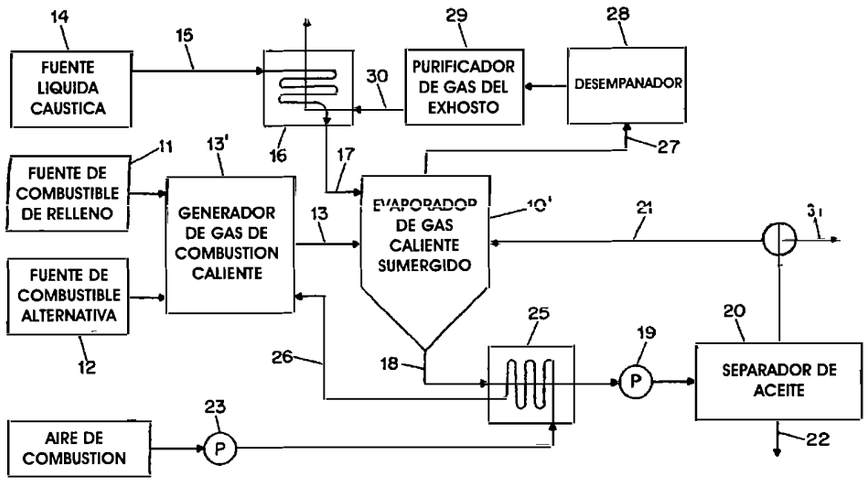
14
FUENTE LIQUIDA CAUSTICA
15
29
PURIFICADOR DE GAS DEL EXHOSTO
28
DESEMPANADOR
30
27
11
FUENTE DE COMBUSTIBLE DE RELLENO
13'
GENERADOR DE GAS DE COMBUSTION CALIENTE
16
17
13
EVAPORADOR DE GAS CALIENTE SUMERGIDO
10'
21
31
FUENTE DE COMBUSTIBLE ALTERNATIVA
12
25
19
20
SEPARADOR DE ACEITE
26
18
P
23
AIRE DE COMBUSTION
P
22

FIG 3

PATENT COOPERATION TREATY

From the
INTERNATIONAL PRELIMINARY EXAMINING AUTHORITY

To.
PAUL A. RAGUSA
BAKER BOTTS L L.P.
30 ROCKEFELLER PLAZA
NEW YORK, NY 10112-4498

# PCT

WRITTEN OPINION

**(PCT Rule 66)**

Date of Mailing *(day/month/year)* 26 NOV 2004

REPLY DUE

within 1 months/days from the above date of mailing

| Applicant's or agent's file reference | | |
|---|---|---|
| 35355-PCT | | |
| *International application No.* | International filing date *(day/month/year)* | Priority date *(day/month/year)* |
| PCT/US03/27517 | 02 September 2003 (02.09.2003) | 04 September 2002 (04.09.2002) |

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

IPC(7): B01D 1/14, 3/34, C01D 3/06; C22B 26/10. and US Cl.: 159/16.1, 16.2, 29, 47.3, DIG.2, DIG.34; 203/49, 100; 210/170; 261/77, 128, DIG.7; 423/183.

*Applicant*

EMCON/OWT, INC.

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1. This written opinion is the first (first, etc.) drawn by this International Preliminary Examining Authority.

2. This opinion contains indications relating to the following items:

- I ☒ Basis of the opinion
- II ☐ Priority
- III ☐ Non-establishment of opinion with regard to novelty, inventive step and industrial applicability
- IV ☐ Lack of unity of invention
- V ☒ Reasoned statement under Rule 66.2 (a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement
- VI ☐ Certain documents cited
- VII ☐ Certain defects in the international application
- VIII ☒ Certain observations on the international application

3 The applicant is hereby **invited to reply** to this opinion.

When? See the time limit indicated above. ~~The applicant may, before the expiration of that time limit, request this Authority to grant an extension. See rule 66.2(d).~~

How? By submitting a written reply, accompanied, where appropriate, by amendments, according to Rule 66 3. For the form and the language of the amendments, see Rules 66.8 and 66.9.

Also For an additional opportunity to submit amendments, see Rule 66.4
For the examiner's obligation to consider amendments and/or arguments, see Rule 66.4 *bis*
For an informal communication with the examiner, see Rule 66 6

**If no reply is filed,** the international preliminary examination report will be established on the basis of this opinion.

4 The final date by which the international preliminary examination report must be established according to Rule 69.2 is: 04 January 2005 (04.01.2005)


Name and mailing address of the IPEA/US
Mail Stop PCT, Attn: IPEA/US
Commissioner for Patents
P.O. Box 1450
Alexandria, Virginia 22313-1450
Facsimile No. (703)305-3230

Authorized officer
Virginia Manoharan
Telephone No. 703-308-0651

## I. Basis of the opinion

1. With regard to the **elements** of the international application:*

☒ the international application as originally filed

☒ the description:
pages 1-16, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.

☒ the claims:
pages 17-19, as originally filed
pages NONE, as amended (together with any statement) under Article 19
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.

☒ the drawings:
pages 1-3, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.

☐ the sequence listing part of the description:
pages NONE, as originally filed
pages NONE, filed with the demand
pages NONE, filed with the letter of _____.

2. With regard to the **language**, all the elements marked above were available or furnished to this Authority in the language in which the international application was filed, unless otherwise indicated under this item.
These elements were available or furnished to this Authority in the following language _____ which is:

☐ the language of a translation furnished for the purposes of international search (under Rule23.1(b)).

☐ the language of publication of the international application (under Rule 48.3(b)).

☐ the language of the translation furnished for the purposes of international preliminary examination(under Rules 55.2 and/or 55.3).

3. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the written opinion was drawn on the basis of the sequence listing:

☐ contained in the international application in printed form.

☐ filed together with the international application in computer readable form.

☐ furnished subsequently to this Authority in written form.

☐ furnished subsequently to this Authority in computer readable form.

☐ The statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.

☐ The statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.

4. ☒ The amendments have resulted in the cancellation of:

☒ the description, pages NONE

☒ the claims, Nos. NONE

☒ the drawings, sheets/fig NONE

5. ☐ This opinion has been drawn as if (some of) the amendments had not been made, since they have been considered to go beyond the disclosure as filed, as indicated in the Supplemental Box (Rule 70.2(c)).

** Replacement sheets which have been furnished to the receiving Office in response to an invitation under Article 14 are referred to in this opinion as "originally filed."*

V. Reasoned statement under Rule 66.2(a)(ii) with regard to novelty, inventive step or industrial applicability; citations and explanations supporting such statement

1. STATEMENT

| | | |
|---|---|---|
| Novelty (N) | Claims 1-19 | YES |
| | Claims NONE | NO |
| Inventive Step (IS) | Claims NONE | YES |
| | Claims 1-19 | NO |
| Industrial Applicability (IA) | Claims 1-19 | YES |
| | Claims NONE | NO |

2. CITATIONS AND EXPLANATIONS

Claims 1-4, 6-16 and 19 lack an inventive step under PCT Article 33(3) as being obvious over DUESEL, JR. , in view of HAMMOND

DUESEL, JR ,does not positively disclose the claimed method of supplying spent caustic effluent to a submerged combustion gas evaporator, but otherwise discloses all the process or method steps as claimed. See Figs. 1-3. It would have been obvious to one of skill in the art, at the time the invention was made, to supply a caustic effluent to a submerged combustion gas evaporator as such is suggested by HAMMOND at page 1, lines 1-30. That is, HAMMOND suggests a method of concentration of caustic soda or other suitable material by submerged combustion.

Claim 18 lacks an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately preceding paragraph and further in view of EWAN et al or ABDEL MALEK et al.

It would have been obvious, at the time the invention was made, to incorporate an air stripping method to the process or method of DUESEL, JR., as modified by HAMMOND, in order to remove dissolved contaminants before discharging material or prior further treatment of fluid material as taught at column 7, lines 27-35 of ABDEL MALEK et al. Note further the advantages to be derived with air stripping as taught at column 24, lines 45-68 by EWAN et al.

Claims 5 and 17 lack an inventive step under PCT Article 33(3) as being obvious over the prior art as applied in the immediately further preceding paragraph and further in view of ANDERSON.

ANDERSON is applied for its teaching that industrial waste water is processed by a submerged combustion evaporator and carbon dioxide in the combustion gases supplied to the waste water is sequestered by calcium hydroxide which has been added to produce calcium carbonate which is then separated from the waste stream.

Claims 1-19 meet the criteria set out in PCT Article 33(4), and thus have industrial applicability because the subject matter claimed can be made or used in industry.

---------- NEW CITATIONS ----------

## VIII. Certain observations on the international application

The following observations on the clarity of the claims, description, and drawings or on the questions whether the claims are fully supported by the description, are made:

Claim 4 is objected to under PCT Rule 66.2(a)(v) as lacking clarity under PCT Article 6 because claim 4 is indefinite for the following reason(s):
Claim 4, as recited, appears to be in improper Markush language

**Supplemental Box**
(To be used when the space in any of the preceding boxes is not sufficient)

TIME LIMIT:
The time limit set for response to a Written Opinion may not be extended. 37 CFR 1.484(d). Any response received after the expiration of the time limit set in the Written Opinion will not be considered in preparing the International Preliminary Examination Report

# PATENT COOPERATION TREATY

# PCT

## INTERNATIONAL SEARCH REPORT

**(PCT Article 18 and Rules 43 and 44)**

| Applicant's or agent's file reference 35355-PCT | **FOR FURTHER ACTION** | see Notification of Transmittal of International Search Report (Form PCT/ISA/220) as well as, where applicable, item 5 below |
|---|---|---|
| International application No. PCT/US03/27517 | International filing date (*day/month/year*) 02 September 2003 (02.09.2003) | (Earliest) Priority Date (*day/month/year*) 04 September 2002 (04.09.2002) |
| Applicant EMCON/OWT, INC. | | |

This international search report has been prepared by this International Searching Authority and is transmitted to the applicant according to Article 18. A copy is being transmitted to the International Bureau.

This international search report consists of a total of 2 sheets.

☒ It is also accompanied by a copy of each prior art document cited in this report.

1. **Basis of the Report**
   a. With regard to the **language**, the international search was carried out on the basis of the international application in the language in which it was filed, unless otherwise indicated under this item.
   ☐ the international search was carried out on the basis of a translation of the international application furnished to this Authority (Rule 23.1(b)).
   b. With regard to any **nucleotide and/or amino acid sequence** disclosed in the international application, the international search was carried out on the basis of the sequence listing:
   ☐ contained in the international application in written form.
   ☐ filed together with the international application in computer readable form.
   ☐ furnished subsequently to this Authority in written form.
   ☐ furnished subsequently to this Authority in computer readable form.
   ☐ the statement that the subsequently furnished written sequence listing does not go beyond the disclosure in the international application as filed has been furnished.
   ☐ the statement that the information recorded in computer readable form is identical to the written sequence listing has been furnished.
2. ☐ **Certain claims were found unsearchable** (See Box I).
3. ☐ **Unity of invention is lacking** (See Box II).
4. With regard to the **title**,
   ☒ the text is approved as submitted by the applicant.
   ☐ the text has been established by this Authority to read as follows:

5. With regard to the **abstract**,
   ☐ the text is approved as submitted by the applicant.
   ☒ the text has been established, according to Rule 38.2(b), by this Authority as it appears in Box III. The applicant may, within one month from the date of mailing of this international search report, submit comments to this Authority.

6. The figure of the **drawings** to be published with the abstract is Figure No. 1
   ☒ as suggested by the applicant. ☐ None of the figures
   ☐ because the applicant failed to suggest a figure.
   ☐ because this figure better characterizes the invention.

# INTERNATIONAL SEARCH REPORT

International application No.

PCT/US03/27517

**Box III TEXT OF THE ABSTRACT (Continuation of Item 5 of the first sheet)**

The technical features mentioned in the abstract do not include a reference sign between parentheses (PCT Rule 8.1(d)).

NEW ABSTRACT

A method for treatment of caustic effluents , a spent caustic refinery effluent from a source (14) is supplied to a submerged combustion gas evaporator (10) in which hot combustion gas containing carbondioxide is injected into the caustic liquid to concentrate the liquid and convert a hydroxide constituent to a carbonate. Where the caustic effluent is from a petroleum refinery, oil in the waste liquid is separated from the aquoeus constituent in an oil separator (20) before, during or after concentration.

# INTERNATIONAL SEARCH REPORT

International application No.
PCT/US03/27517

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : B01D 1/14, 3/34; C01D 3/06; C22B 26/10.
US CL : 159/16.1, 16.2, 29, 47.3, DIG.2, DIG.34; 203/49, 100; 210/170; 261/77, 128, DIG.7; 423/183.

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)
U.S. : 159/16.1, 16.2, 29, 44, 47.3, DIG.2, DIG.34; 202/234; 203/2, 49, 100; 210/170; 261/77, 128, DIG.7; 423/183.

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
NONE

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

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

| Category * | Citation of document, with indication, where appropriate, of the relevant passages | Relevant to claim No. |
|---|---|---|
| Y | US 5,342,482 A (DUESEL, JR) 30 August 1994, column 2, lines 49-56, column 3, lines 52-60 and Figs. 1-3. | 1-4, 6-16, 19 |
| Y | US 1,668,504 A (HAMMOND) 01 May 1928, page 1, lines 1-30, page 3, lines 8-13. | 1-4, 6-16, 19 |
| Y | US 5,642,630 A (ABDELMALEK et al ) ) 01 July 1997, column 7, lines 27-35. | 18 |
| Y | US 4,188,291 A (ANDERSON) 12 February 1980, columns 7-8. | 5, 17 |
| Y | US 4,141,701 A (EWAN et al ) 27 February 1979, column 24, lines 45-68. | 18 |
| A | US 3,275,062 A (WILLIAMS) 27 September 1966, see entire document. | 1-19 |

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

* Special categories of cited documents.

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier application or patent published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"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

"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

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

| Date of the actual completion of the international search | Date of mailing of the international search report |
|---|---|
| 19 April 2004 (19.04.2004) | 07 MAY 2004 |
| Name and mailing address of the ISA/US<br>Mail Stop PCT, Attn: ISA/US<br>Commissioner for Patents<br>P.O. Box 1450<br>Alexandria, Virginia 22313-1450<br>Facsimile No. (703)305-3230 | Authorized officer<br>Virginia Manoharan<br>Telephone No. 703-308-0651 |

Form PCT/ISA/210 (second sheet) (July 1998)

## NOTES TO FORM PCT/ISA/220

These Notes are intended to give the basic instructions concerning the filing of amendments under Article 19. The Notes are based on the requirements of the Patent Cooperation Treaty, the Regulations and the Administrative Instructions under that Treaty. In case of discrepancy between these Notes and those requirements, the latter are applicable. For more detailed information, see also the *PCT Applicant's Guide*, a publication of WIPO.

In these Notes, "Article," "Rule" and "Section" refer to the provisions of the PCT, the PCT Regulations and the PCT Administrative Instructions, respectively.

## INSTRUCTIONS CONCERNING AMENDMENTS UNDER ARTICLE 19

The applicant has, after having received the international search report, one opportunity to amend the claims of the international application. It should however be emphasized that, since all parts of the international application (claims, description and drawings) may be amended during the international preliminary examination procedure, there is usually no need to file amendments of the claims under Article 19 except where, e.g. the applicant wants the latter to be published for the purposes of provisional protection or has another reason for amending the claims before international publication. *Furthermore, it should be emphasized that provisional protection is available in some States only.*

**What parts of the international application may be amended ?**

Under Article 19, only the claims may be amended.

During the international phase, the claims may also be amended (or further amended) under Article 34 before the International Preliminary Examining Authority. The description and drawings may only be amended under Article 34 before the International Preliminary Examining Authority.

Upon entry into the national phase, all parts of the international application may be amended under Article 28 or, where applicable, Article 41.

**When ?** Within 2 months from the date of transmittal of the international search report or 16 months from the priority date, whichever time limit expires later. It should be noted, however, that the amendments will be considered as having been received on time if they are received by the International Bureau after the expiration of the applicable time limit but before the completion of the technical preparations for international publication (Rule 46.1).

**Where not to file the amendments ?**

The amendments may only be filed with the International Bureau and not with the receiving Office or the International Searching Authority (Rule 46.2).

Where a demand for international preliminary examination has been/is filed, see below.

**How ?** Either by cancelling one or more entire claims, by adding one or more new claims or by amending the text of one or more of the claims as filed.

A replacement sheet must be submitted for each sheet of the claims which, on account of an amendment or amendments, differs from the sheet originally filed.

All the claims appearing on a replacement sheet must be numbered in Arabic numerals. Where a claim is cancelled, no renumbering of the other claims is required. In all cases where claims are renumbered, they must be renumbered consecutively (Administrative Instructions, Section 205(b)).

**The amendments must be made in the language in which the international application is to be published.**

**What documents must/may accompany the amendments ?**

**Letter (Section 205(b)):**

The amendments must be submitted with a letter.

The letter will not be published with the international application and the amended claims. It should not be confused with the "Statement under Article 19(1)" (see below, under "Statement under Article 19(1)").

**The letter must be in English or French, at the choice of the applicant. However, if the language of the international application is English, the letter must be in English; if the language of the international application is French, the letter must be in French.**

The letter must indicate the differences between the claims as filed and the claims as amended. It must, in particular, indicate, in connection with each claim appearing in the international application (it being understood that identical indications concerning several claims may be grouped), whether

(i) the claim is unchanged;

(ii) the claim is cancelled;

(iii) the claim is new;

(iv) the claim replaces one or more claims as filed;

(v) the claim is the result of the division of a claim as filed.

**The following examples illustrate the manner in which amendments must be explained in the accompanying letter:**

1. [Where originally there were 48 claims and after amendment of some claims there are 51]
"Claims 1 to 29, 31, 32, 34, 35, 37 to 48 replaced by amended claims bearing the same numbers; claims 30, 33 and 36 unchanged; new claims 49 to 51 added."

2 [Where originally there were 15 claims and after amendment of all claims there are 11]:
"Claims 1 to 15 replaced by amended claims 1 to 11."

3. [Where originally there were 14 claims and the amendments consist in cancelling some claims and in adding new claims]:
"Claims 1 to 6 and 14 unchanged, claims 7 to 13 cancelled; new claims 15, 16 and 17 added." or
"Claims 7 to 13 cancelled; new claims 15, 16 and 17 added; all other claims unchanged."

4. [Where various kinds of amendments are made]:
"Claims 1-10 unchanged, claims 11 to 13, 18 and 19 cancelled; claims 14, 15 and 16 replaced by amended claim 14; claim 17 subdivided into amended claims 15, 16 and 17; new claims 20 and 21 added."

**"Statement under Article 19(1)" (Rule 46.4)**

The amendments may be accompanied by a statement explaining the amendments and indicating any impact that such amendments might have on the description and the drawings (which cannot be amended under Article 19(1)).

The statement will be published with the international application and the amended claims.

**It must be in the language in which the international application is to be published.**

It must be brief, not exceeding 500 words if in English or if translated into English.

It should not be confused with and does not replace the letter indicating the differences between the claims as filed and as amended. It must be filed on a separate sheet and must be identified as such by a heading, preferably by using the words "Statement under Article 19(1)."

It may not contain any disparaging comments on the international search report or the relevance of citations contained in that report. Reference to citations, relevant to a given claim, contained in the international search report may be made only in connection with an amendment of that claim.

***Consequence if a demand for international preliminary examination has already been filed***

If, at the time of filing any amendments and any accompanying statement, under Article 19, a demand for international preliminary examination has already been submitted, the applicant must preferably, at the time of filing the amendments (and any statement) with the International Bureau, also file with the International Preliminary Examining Authority a copy of such amendments (and of any statement) and, where required, a translation of such amendments for the procedure before that Authority (see Rules 55.3(a) and 62.2, first sentence). For further information, see the Notes to the demand form (PCT/IPEA/401).

**Consequence with regard to translation of the international application for entry into the national phase**

The applicant's attention is drawn to the fact that, upon entry into the national phase, a translation of the claims as amended under Article 19 may have to be furnished to the designated/elected Offices, instead of, or in addition to, the translation of the claims as filed.

For further details on the requirements of each designated/elected Office, see the *PCT Applicant's Guide*, Volume II.

*The demand must be filed directly with the competent International Preliminary Examining Authority or, if two or more Authorities are competent, with the one chosen by the applicant. The full name or two-letter code of that Authority may be indicated by the applicant on the line below:*

**IPEA/** US

# PCT

**CHAPTER II**

## DEMAND

under Article 31 of the Patent Cooperation Treaty:
The undersigned requests that the international application specified below be the subject of international preliminary examination according to the Patent Cooperation Treaty and hereby elects all eligible States (except where otherwise indicated).

For International Preliminary Examining Authority use only

| Identification of IPEA | Date of receipt of DEMAND |
|---|---|
| | |

| **Box No. I IDENTIFICATION OF THE INTERNATIONAL APPLICATION** | | Applicant's or agent's file reference: 35355-PCT |
|---|---|---|
| International application No.: PCT/US03/27517 | International filing date *(day/month/year)*: 02 September 2003 (02.09.03) | (Earliest) Priority date *(day/month/year)*: 04 September 2002 (04.09.02) |

Title of invention: TREATMENT OF SPENT CAUSTIC REFINERY EFFLUENTS

**Box No. II APPLICANT(S)**

| Name and address: *(Family name followed by given name, for a legal entity, full official designation. The address must include postal code and name of country.)* | Telephone No. |
|---|---|
| EMCON/OWT, INC.<br>6910 Treeline Drive, Suite F<br>Brecksville, OH 44141<br>US | Facsimile No. |
| | Teleprinter No. |
| | Applicant's registration No. with the Office |

| State *(that is, country)* of nationality: | State *(that is, country)* of residence: |
|---|---|
| US | US |

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

DUESEL, Jr., Bernard F.
4 Partridge Lane
Goshen, NY 10924
US

| State *(that is, country)* of nationality: | State *(that is, country)* of residence: |
|---|---|
| US | US |

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

GIBBONS, John P.
4 Harold Avenue
Cornwall, NY 12518
US

| State *(that is, country)* of nationality: | State *(that is, country)* of residence: |
|---|---|
| US | US |

☒ Further applicants are indicated on a continuation sheet.

International application No.
PCT/US03/27517

**Continuation of Box No. II APPLICANT(S)**

*If none of the following sub-boxes is used, this sheet should not be included in the demand.*

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

RUTSCH, Michael J.
8329 South 75th East Avenue
Tulsa, OK 74133
US

| State *(that is, country)* of nationality: | State *(that is, country)* of residence: |
|---|---|
| US | US |

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

| State *(that is, country)* of nationality: | State *(that is, country)* of residence: |
|---|---|
| | |

Name and address: *(Family name followed by given name, for a legal entity, full official designation. The address must include postal code and name of country.)*

| State *(that is, country)* of nationality: | State *(that is, country)* of residence: |
|---|---|
| | |

Name and address: *(Family name followed by given name, for a legal entity, full official designation. The address must include postal code and name of country.)*

| State *(that is, country)* of nationality: | State *(that is, country)* of residence: |
|---|---|
| | |

☐ Further applicants are indicated on another continuation sheet.

Sheet No. 3.

International application No.
PCT/US03/27517

**Box No. III AGENT OR COMMON REPRESENTATIVE; OR ADDRESS FOR CORRESPONDENCE**

The following person is [X] agent [ ] common representative

and [X] has been appointed earlier and represents the applicant(s) also for international preliminary examination.

[ ] is hereby appointed and any earlier appointment of (an) agent(s)/common representative is hereby revoked.

[ ] is hereby appointed, specifically for the procedure before the International Preliminary Examining Authority, in addition to the agent(s)/common representative appointed earlier.

Name and address: *(Family name followed by given name; for a legal entity, full official designation. The address must include postal code and name of country.)*

RAGUSA, Paul A.
BAKER BOTTS L.L.P.
30 Rockefeller Plaza
New York, NY 10112
US

Telephone No.
212-408-2588

Facsimile No.
212-259-2588

Teleprinter No.

Agent's registration No. with the Office
38,587

[ ] **Address for correspondence:** Mark this check-box where no agent or common representative is/has been appointed and the space above is used instead to indicate a special address to which correspondence should be sent.

**Box No. IV BASIS FOR INTERNATIONAL PRELIMINARY EXAMINATION**

**Statement concerning amendments:***

1. The applicant wishes the international preliminary examination **to start on the basis of:**

[X] the international application as originally filed

the description
[ ] as originally filed
[ ] as amended under Article 34

the claims
[ ] as originally filed
[ ] as amended under Article 19 (together with any accompanying statement)
[ ] as amended under Article 34

the drawings
[ ] as originally filed
[ ] as amended under Article 34

2. [ ] The applicant wishes any amendment to the claims under Article 19 to be considered as reversed.

3. [ ] The applicant wishes the start of the international preliminary examination **to be postponed** until the expiration of 20 months from the priority date unless the International Preliminary Examining Authority receives a copy of any amendments made under Article 19 or a notice from the applicant that he does not wish to make such amendments (Rule 69.1(d)). *(This check-box may be marked only where the time limit under Article 19 has not yet expired.)*

* Where no check-box is marked, international preliminary examination will start on the basis of the international application as originally filed or, where a copy of amendments to the claims under Article 19 and/or amendments of the international application under Article 34 are received by the International Preliminary Examining Authority before it has begun to draw up a written opinion or the international preliminary examination report, as so amended.

**Language for the purposes of international preliminary examination:** English

[X] which is the language in which the international application was filed.
[ ] which is the language of a translation furnished for the purposes of international search.
[ ] which is the language of publication of the international application.
[ ] which is the language of the translation (to be) furnished for the purposes of international preliminary examination.

**Box No. V ELECTION OF STATES**

The applicant hereby **elects all eligible States** *(that is, all States which have been designated and which are bound by Chapter II of the PCT)*

excluding the following States which the applicant wishes **not to elect:**

Sheet No. 4.

International application No.
PCT/US03/27517

**Box No. VI CHECK LIST**

| The demand is accompanied by the following elements, in the language referred to in Box No. IV, for the purposes of international preliminary examination: | | | For International Preliminary Examining Authority use only: received | not received |
|---|---|---|---|---|
| 1. translation of international application | : | sheets | ☐ | ☐ |
| 2. amendments under Article 34 | : | sheets | ☐ | ☐ |
| 3. copy (or, where required, translation) of amendments under Article 19 | : | sheets | ☐ | ☐ |
| 4. copy (or, where required, translation) of statement under Article 19 | : | sheets | ☐ | ☐ |
| 5. letter | : | sheets | ☐ | ☐ |
| 6. other *(specify)* | : | sheets | ☐ | ☐ |

The demand is also accompanied by the item(s) marked below:

1. ☒ fee calculation sheet
2. ☐ original separate power of attorney
3. ☐ original general power of attorney
4. ☐ copy of general power of attorney; reference number, if any:
5. ☐ statement explaining lack of signature
6. ☐ sequence listing in computer readable form
7. ☒ other *(specify)*: Transmittal Letter and a check for $748.00

**Box No. VII SIGNATURE OF APPLICANT, AGENT OR COMMON REPRESENTATIVE**

*Next to each signature, indicate the name of the person signing and the capacity in which the person signs (if such capacity is not obvious from reading the demand).*

Paul A. Ragusa (Agent)

**For International Preliminary Examining Authority use only**

1. Date of actual receipt of DEMAND:
2. Adjusted date of receipt of demand due to CORRECTIONS under Rule 60.1(b):
3. ☐ The date of receipt of the demand is AFTER the expiration of 19 months from the priority date and item 4 or 5, below, does not apply. ☐ The applicant has been informed accordingly.
4. ☐ The date of receipt of the demand is WITHIN the period of 19 months from the priority date as extended by virtue of Rule 80.5.
5. ☐ Although the date of receipt of the demand is after the expiration of 19 months from the priority date, the delay in arrival is EXCUSED pursuant to Rule 82.

**For International Bureau use only**

Demand received from IPEA on:

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**CHAPTER II**

# PCT

## FEE CALCULATION SHEET

**Annex to the Demand**

| | |
|---|---|
| International application No. PCT/US03/27517 | For International Preliminary Examining Authority use only |
| Applicant's or agent's file reference 35355-PCT | Date stamp of the IPEA |

Applicant

EMCON/OWT, INC.

### CALCULATION OF PRESCRIBED FEES

| | | |
|---|---|---|
| 1. Preliminary examination fee ..... | 600.00 P | |
| 2. Handling fee *(Applicants from certain States are entitled to a reduction of 75% of the handling fee. Where the applicant is (or all applicants are) so entitled, the amount to be entered at H is 25% of the handling fee.)* ..... | 148.00 H | |
| 3. Total of prescribed fees Add the amounts entered at P and H and enter total in the TOTAL box ..... | 748.00 TOTAL | |

### MODE OF PAYMENT

| | | | |
|---|---|---|---|
| ☐ | authorization to charge deposit account with the IPEA (see below) | ☐ | cash |
| ☒ | cheque | ☐ | revenue stamps |
| ☐ | postal money order | ☐ | coupons |
| ☐ | bank draft | ☐ | other *(specify)*: |

### AUTHORIZATION TO CHARGE (OR CREDIT) DEPOSIT ACCOUNT

*(This mode of payment may not be available at all IPEAs)*

IPEA/ US

☐ Authorization to charge the total fees indicated above.

☒ *(This check-box may be marked only if the conditions for deposit accounts of the IPEA so permit)* Authorization to charge any deficiency or credit any overpayment in the total fees indicated above.

Deposit Account No.: 02-4377

Date: 01 April 2004

Name: Paul A. Ragusa

Signature:

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# REQUISITOS NECESARIOS PARA PRESENTACION Y AGILIZACION DEL TRAMITE

| | USUARIO | RADICADOR |
|---|---|---|
| PATENTE DE INVENCIÓN MODELO DE UTILIDAD | ( ) | ( ) |
| - Indicación de que se solicita la concesión de una patente. | ( ) | ( ) |
| - Datos de identificación del solicitante o de la persona que se presenta la solicitud. | ( ) | ( ) |
| - Descripción de la invención. | ( ) | ( ) |
| - Dibujos de ser estos pertinentes. | ( ) | ( ) |
| - Comprobante de Pago de las tasas establecidas. | | |
| COMPLETA ( ) INCOMPLETA | ( ) | ( ) |
| DISEÑO INDUSTRIAL ( ) | | |
| - Indicación de que se solicita el registro de Diseño Industrial | ( ) | ( ) |
| - Datos de identificación del solicitante o de la persona que presenta | ( ) | ( ) |
| - la solicitud. | | |
| - Representación gráfica o fotográfica del Diseño Industrial o muestra del Material que incorpora el diseño. | ( ) | ( ) |
| Comprobante de pago de las tasas establecidas | ( ) | ( ) |
| COMPLETA ( ) INCOMPLETA | ( ) | ( ) |
| ESQUEMA DE TRAZADO ( ) | | |
| - Indicación de que solicita el registro de un Esquema de trazado. | ( ) | ( ) |
| - Datos de identificación del solcitante o de la persona que presenta La solicitud. | ( ) | ( ) |
| - Representación gráfica del esquema de trazado | ( ) | ( ) |
| - Comprobante de pago de las tasas establecidas. | ( ) | ( ) |
| COMPLETA ( ) INCOMPLETA | ( ) | ( ) |

Firma Responsable:

Fecha:

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Dependencia: 2020 DIVISION DE NUEVAS CREACIONES

Industria y Comercio
SUPERINTENDENCIA

2020
Bogotá, D.C.,

**Doctora:**
**DILIA MARIA RODRÍGUEZ D' ALEMAN**
Apoderado
**EMCON / OWT, INC.**

**Oficio N°** 12527

| | | |
|---|---|---|
| **ASUNTO:** | Radicación N° | 05-28020 |
| | Solicitud internacional | PCT/US2003/027517 |
| | Fecha inicio fase nacional | 04/04/2005 |
| | Trámite | 011 |
| | Evento | 379 |
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| | Folios | 001 |

Como resultado del examen de forma, realizado con fundamento en el artículo 38 de la Decisión 486 de la Comisión de la Comunidad Andina, artículo 27 del Tratado de Cooperación en Materia de Patentes (PCT), regla 51 bis del Reglamento y Circular Única de la Superintendencia de Industria y Comercio, se le comunica que:

- Si fuere el caso, el solicitante debe adjuntar la traducción de los anexos del reporte de examen preliminar internacional (Art. 36.2.b) del tratado PCT).

- La solicitud no contiene la copia del documento en que consta la cesión del derecho a la patente del inventor al solicitante o a su causante. (Art. 26-k) Decisión 486).

- La solicitud no contiene el poder debidamente otorgado, con el lleno de los requisitos exigidos por los artículos 63 y subsiguientes del Código de Procedimiento Civil. Adicionalmente, deberá presentar el documento que acredita la existencia y representación legal de la sociedad solicitante, cuando ésta fuere persona jurídica.

En cumplimiento de lo dispuesto por el artículo 39 de la Decisión 486, se le requiere para que dentro del término de dos meses siguientes a la fecha de notificación del presente oficio, complete los requisitos anteriormente enunciados. En caso de no dar cumplimiento al presente requerimiento, la solicitud se considerará abandonada y perderá su prelación.

Notifíquese el presente oficio conforme a lo dispuesto en el numeral 5.2, literal e), del capítulo quinto del título primero de la Circular Única N° 10 de 2001.

**ALIX CARMENZA CESPEDES DE VERGEL**
Jefe de la División de Nuevas Creaciones.

El anterior requerimiento fue notificado por fijación en lista de fecha, 08 ABR. 2005

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Carrera 13 No. 27-00 Piso 5 y 10
Conmutador: 334 12 21-2-3-4
Fax: 281 31 25 e-mail: info@sic.gov.co
Santa Fe de Bogotá D.C. - Colombia