



- (51) **International Patent Classification:**
B01D 3/02 (2006.01)
- (21) **International Application Number:**
PCT/IN2014/000448
- (22) **International Filing Date:**
4 July 2014 (04.07.2014)
- (25) **Filing Language:** English
- (26) **Publication Language:** English
- (30) **Priority Data:**
2620/MUM/2013 8 August 2013 (08.08.2013) IN
- (71) **Applicant:** PRAJ INDUSTRIES LIMITED [IN/IN];
PRAJ Tower, 274-275, Bhumkar Chowk - Hinjewadi
Road, Hinjewadi, Pune - 411 057 (IN).
- (72) **Inventors:** GHANSHAM BABURAO DESHPANDE;
PRAJ INDUSTRIES LIMITED, PRAJ Tower, 274-275,
Bhumkar Chowk - Hinjewadi Road, Hinjewadi, Pune - 411
057 (IN). SHRIKANT SUBHASH RATHI; PRAJ IN-
DUSTRIES LIMITED, PRAJ Tower, 274-275, Bhumkar
Chowk - Hinjewadi Road, Hinjewadi, Pune - 411 057 (IN).
DEVDATTA KRISHNA DESHPANDE; PRAJ INDUS-
TRIES LIMITED, PRAJ Tower, 274-275, Bhumkar
Chowk - Hinjewadi Road, Hinjewadi, Pune - 411 057 (IN).
PRASANNA SHAM PAI; PRAJ INDUSTRIES LIM-
ITED, PRAJ Tower, 274-275, Bhumkar Chowk - Hinjew-
adi Road, Hinjewadi, Pune - 411 057 (IN). PANDURANG
RAM SHINDE; PRAJ INDUSTRIES LIMITED, PRAJ
Tower, 274-275, Bhumkar Chowk - Hinjewadi Road, Hin-
jewadi, Pune - 411 057 (IN).
- (74) **Agent:** PARAG KINGE; PRAJ INDUSTRIES LIMITED,
PRAJ Tower, 274-275, Bhumkar Chowk - Hinjewadi
Road, Hinjewadi, Pune - 411 057 (IN).
- (81) **Designated States** (*unless otherwise indicated, for every
kind of national protection available*): AE, AG, AL, AM,
AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY,
BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM,
DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT,
HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR,
KZ, LA, LC, LK, LR, LS, LT, LU, LY, MA, MD, ME,
MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ,
OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA,
SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM,
TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM,
ZW.
- (84) **Designated States** (*unless otherwise indicated, for every
kind of regional protection available*): ARIPO (BW, GH,
GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, SZ, TZ,
UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ,
TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK,
EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV,
MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM,
TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW,
KM, ML, MR, NE, SN, TD, TG).
- Declarations under Rule 4.17:**
- *as to applicant's entitlement to apply for and be granted a
patent (Rule 4.17(ii))*
- Published:**
- *without international search report and to be republished
upon receipt of that report (Rule 48.2(g))*

(54) **Title:** PRODUCTION OF ETHANOL BY EVAPORATIVE DISTILLATION

(57) **Abstract:** A process for producing ethanol by evaporative distillation is disclosed, wherein fermented wash is first evaporated in an evaporator to generate a concentrated ethanol stream. The remaining part of said wash is then further purified in a mash column to isolate remaining ethanol from said wash. In this process as a large part of fermented wash is removed in evaporation process, the load on the mash column is significantly less and hence small size of mash column is required.



TITLE: PRODUCTION OF ETHANOL BY EVAPORATIVE DISTILLATION**FIELD OF THE INVENTION**

The invention relates to a process for the preparation of ethanol using a technique of evaporation distillation, wherein significantly less amount of heat energy is required compared with a process not employing the process of the invention disclosed herein.

BACKGROUND

Many alcoholic beverages are produced comprising one or more of the steps of: [1] producing ethanol by fermentation of a carbohydrate-rich feedstock to produce a fermented wash having concentration of about 2 to 23% alcohol by volume [ABV]; [2] distilling the product of fermentation at elevated temperatures to produce ethanol products like rectified spirit, neutral spirit or absolute alcohol; and [3] aging the ethanol spirits until it possesses desired flavour, aroma, and colour characteristics. The commercial production of alcohol by distillation has been in widespread operation for many centuries.

The distillation is a known technique for purification of a liquid substance and involves vaporizing the substance at its boiling point, condensing the vapour and collecting the purified form as a condensate. Distillation is useful for separating a mixture when the components have different boiling points. Several kinds of distillation techniques for binary or multi-component mixtures are described and practiced in the art, for example: (1) simple, (2) vacuum or reduced pressure, (3) fractional and (4) steam distillation.

In all techniques available for distillation of ethanol from fermented wash the consumption of steam energy per litre of ethanol produced is the critical

measure for economics of the process. Presently, a typical ethanol production process uses about 2 to 3 kilogram of steam per litre of ethanol produced. However, more steam is required if the titre of ethanol in fermented wash is low. There is need of less consumption of steam per litre of ethanol consumed and the invention disclosed herein deals with a solution to the problem of high steam consumption to produce ethanol from fermented wash. The invention presented herein discloses a method of evaporation of fermented wash before distillation having several advantages over conventional distillation methods

BRIEF DESCRIPTION

A process for producing ethanol by evaporative distillation is disclosed comprising: preheating a fermented wash forming a first stream; subjecting said first stream to a degasifying column forming a second stream and a first vapour stream; condensing and collecting said first vapour stream in a collection tank forming a high ethanol stream; subjecting said second stream to a first evaporator forming a second vapour stream and a depleted ethanol stream; condensing said second vapour stream using a second evaporator forming a third stream and collecting it in said collection tank forming said high ethanol stream; supplying a small part of said second vapour stream to said degasifying column as heat source; subjecting said depleted ethanol stream to a distillation column forming a third vapour stream and a spent stream; ~~condensing said third vapour stream using said first evaporator forming a~~ fourth stream and collecting it in said collection tank forming said high ethanol stream; supplying said spent stream to said second evaporator and concentrating it to form a concentrated spent stream and a process condensate stream; and subjecting said high ethanol stream to a rectification process to recover rectified ethanol.

DESCRIPTION OF THE DRAWINGS

Particular examples of methods in accordance with this invention will now be described with reference to accompanying drawing, in which:

FIGURE 1 is an exemplary plan of the invention showing several features that control the process of evaporative distillation of ethanol from a fermented wash. A fermented wash after preheating [2] is fed to a degasifying column [A]. A stream [3] from bottom of said degasifying column is fed to a first evaporator [C], where it gets heated to form next stream [4], which through a heat exchanger [F1] heats stream [1] and then further gets heated in a second heat exchanger [F2] to higher temperature before being sent to a mash column [E], forming a high ethanol stream [8] which further collect into collecting tank [B] via evaporator [C]. The top stream [11] of evaporator [C] is split; and one portion runs said degasifying column [A], while remaining portion is condensed by evaporator [D] forming stream [12], which is collected in tank [B]. The bottom stream [5] from said mash column [E] is fed to evaporator [D] to remove water [7] forming a concentrated spent stream [6]. The vapour stream [9] from column [A] is condensed and collected in tank [B]. The ethanol content of high ethanol stream [10] is about 40% by volume, which is sent to further purification/ rectification.

FIGURE 2 is another exemplary plan of the invention wherein a fermented wash after preheating [2] is split into two streams at a ratio of 70:30, the larger stream [2] is fed to a degasifying column [A], while the smaller stream [13] is directly fed to a mash column [E] keeping the other steps of the process as depicted in FIGURE 1. This by-pass of about 30 % of preheated fermented wash [13] directly to the mash column [E] increases the vapour content of stream [8] leading to increase in evaporation efficiency of the evaporators significantly. This is an optional feature for increasing the efficiency of the process disclosed herein.

DETAILED DESCRIPTION

In one embodiment of the present invention, a fermented wash with ethanol concentration of between 2 to 23% ABV, preferably 6 to 14% ABV is obtained from yeast fermentation of a carbohydrate rich feedstock like grains, cereals, tubers, molasses, sugarcane juice, lignocellulosic materials or synthetic ethanol. This wash is then preheated to about 60°C using a hot vapour stream of first evaporator in a heat exchanger. This preheated wash [first stream] is subjected to a degasifying column to separate gases and an top ethanol rich stream [first vapour stream]. After removal of gases, said ethanol rich stream is condensed and collected in a collection tank. Said degasifying column is heated by a part of top stream [second vapour stream] obtained from said first evaporator. The bottom stream [second stream] of said degasifying column is fed to said first evaporator forming a second vapour stream and a depleted ethanol stream. Another part of said second vapour stream is fed to a second evaporator forming a third stream, which is collected in said collection tank. Simultaneously, said second evaporator concentrates a spent stream from a mash column, separating water for the process recycle. Said depleted ethanol stream from the bottom of said first evaporator is further preheated to about 80°C by energy of said spent stream in a heat exchanger and then fed to said mash column forming a third vapour stream and said spent stream. Said first evaporator is heated by the energy present in said third vapour stream, condensing and collecting it in said collection tank. Said collection tank gives high ethanol stream suitable for further rectification. Said mash column is supplied with heat for energy requirements. Ethanol content of stream from said collection tank is about 30 to 40% by volume.

In another embodiment of the disclosed invention as illustrated in FIGURE 1 the steps of said process are: the fermented wash [1] is preheated with a bottom stream [4] of a first evaporator [C] in a heat exchanger [F1]. This preheated stream [2] is fed to a degasifying column [A]. Herein, gases from [2] are removed along with ethanol vapours [9], which are condensed and

collected in a collection tank [B]. A bottom stream [3] from said degasifying column is then subjected to said first evaporator [C] heating it to form two streams; a top ethanol rich vapour stream [11] and bottom stream [4]. Said vapour stream [11] is split and some part is sent to degasifying column [A] for its running and rest is sent to a second evaporator [D], wherein it is condensed to form stream [12], which is collected in said collection tank [B]. The energy of vapour stream [11] is used to concentrate a spent stream [5] coming from mash column [E] to separate process water for recycle [7]. Said bottom stream [4] is preheated with a heat exchanger [F2] using said spent stream [5] and subjected to distillation in said mash column [E], leading to said vapour stream [8] and said spend stream [5]. Said mash column is heated externally to maintain a desired temperature in the system. This method of efficient use of energy affords about 40% ethanol by volume in collection tank [B], which is subjected to further purification and rectification.

In a different embodiment of the disclosed invention as illustrated in FIGURE 2 the steps of said process are same as per FIGURE 1 except that the preheated stream [2] is divided in a ratio of 70:30. The larger stream [2] is fed to a degasifying column [A], while the smaller stream [13] is directly fed to the mash column [E]. This by-pass of about 30 % of preheated fermented wash [13] directly to the mash column [E] increases the vapour content of stream [8] leading to increase in evaporation efficiency of the evaporators significantly. This is an optional feature for increasing the efficiency of the process disclosed herein.

In yet another embodiment a process for producing ethanol by evaporative distillation is disclosed, wherein fermented wash is first evaporated in an evaporator to generate a concentrated ethanol stream. The remaining part of said wash is then further purified in a mash column to isolate remaining ethanol from said wash. In this process as a large part of fermented wash is removed in evaporation process, the load on the mash column is significantly less and hence small size of mash column is required.

Example provided below gives wider utility of the invention without any limitations as to the variations that may be appreciated by a person skilled in the art. A non-limiting summary of various embodiments is given in the examples and tables, which demonstrate the advantageous and novel aspects of the process disclosed herein. Particular examples of processes in accordance with this invention will now be described with reference to the accompanying drawings

EXAMPLE

In one embodiment of the present invention about 35 KLPD ethanol production plant is operated using the method of evaporative distillation disclosed herein. In a plant fermented wash produced at the rate of about 17 TPH contained about 8% v/v of ethanol. This fermented wash at about 32 °C was preheated to about 58 to 60 °C by a plate type heat exchanger utilizing the excess heat from the concentrated bottom stream of first evaporation unit. Said preheated stream (first stream) was fed into degasifying column to remove the dissolved gases from the fermented wash and produced a first vapour stream and a bottom second stream having concentration of about 8 % V/V of ethanol. Said first vapour stream was condensed to produce high ethanol stream and collected in a collection tank. Said second stream was further subjected to said first evaporator, generating a second vapour stream and a depleted ethanol stream. Here said first evaporator is heat integrated with a mash column downstream. Said second vapour stream from said first evaporator contained about 37% V/V of ethanol, which was condensed to produce high ethanol stream and collected in said collection tank. Said depleted ethanol stream produced in bottom contained about 4.2 % V/V of ethanol which was fed to said mash column. Before entry to said mash column said depleted ethanol stream was preheated in a heat exchanger by excess heat of spent stream of said mash column. Said mash column was

provided required heat from an external source. The third vapour stream from the top of said mash column ran said first evaporator. Said spent stream fed to a second evaporator for concentrating it to contain about 12% total solid by weight. The third vapour stream generated from mash column contained about 26% V/V of ethanol which was condensed to produce high ethanol stream and collected in said collection tank. The said final high ethanol stream from collection tank was subjected to rectification to obtain 95 % V/V of ethanol. This method afforded about 35 KLPD of ethanol from about 17 TPH of fermented wash with consumption of steam at the rate of about 1.3 kilogram per litre of ethanol.

While the invention has been particularly shown and described with reference to embodiments listed in examples, it will be appreciated that several of the above disclosed and other features and functions, or alternatives thereof, may be desirably combined into many other different systems or applications. Also that various presently unforeseen and unanticipated alternatives, modifications, variations, or improvements therein may be subsequently made by those skilled in the art which are also intended to be encompassed by the following claims. Although the invention has been described with reference to specific preferred embodiments, it is not intended to be limited thereto, rather those having ordinary skill in the art will recognize that variations and modifications may be made therein which are within the spirit of the invention and within the scope of the claims.

CLAIMS

1. A process for producing ethanol by evaporative distillation comprising:

- a. preheating a fermented wash forming a first stream;
- b. subjecting said first stream to a degasifying column forming a second stream and a first vapour stream;
- c. condensing and collecting said first vapour stream in a collection tank forming a high ethanol stream;
- d. subjecting said second stream to a first evaporator forming a second vapour stream and a depleted ethanol stream;
- e. condensing said second vapour stream using a second evaporator forming a third stream and collecting it in said collection tank forming said high ethanol stream;
- f. supplying a small part of said second vapour stream to said degasifying column as heat source.
- g. subjecting said depleted ethanol stream to a mash column forming a third vapour stream and a spent stream;
- h. condensing said third vapour stream using said first evaporator forming a fourth stream and collecting it in said collection tank forming said high ethanol stream;
- i. supplying said spent stream to said second evaporator and concentrating it to form a concentrated spent stream and a process condensate stream; and
- j. subjecting said high ethanol stream to a rectification process to recover rectified ethanol.

2. The process of claim 1, wherein said fermented wash is preheated to about 60 °C.
3. The process of claim 1, wherein said degasifying column removes gaseous part of said fermented wash.
4. The process of claim 1, wherein said first evaporator is energized using said third vapour stream.
5. The process of claim 1, wherein said second evaporator is energized using said second vapour stream.
6. The process of claim 1, wherein said mash column is operated using steam energy as necessary.
7. The process of claim 1, wherein said depleted ethanol stream is used to preheat said fermented wash.
8. The process of claim 1, wherein said spent stream is used to preheat said depleted ethanol stream.
9. The process of claim 1, wherein said process condensate stream is recycled to recover process water.
10. The process of claim 1, wherein size of said mash column is reduced to about 50% by of evaporation of said fermented wash prior to distillation.

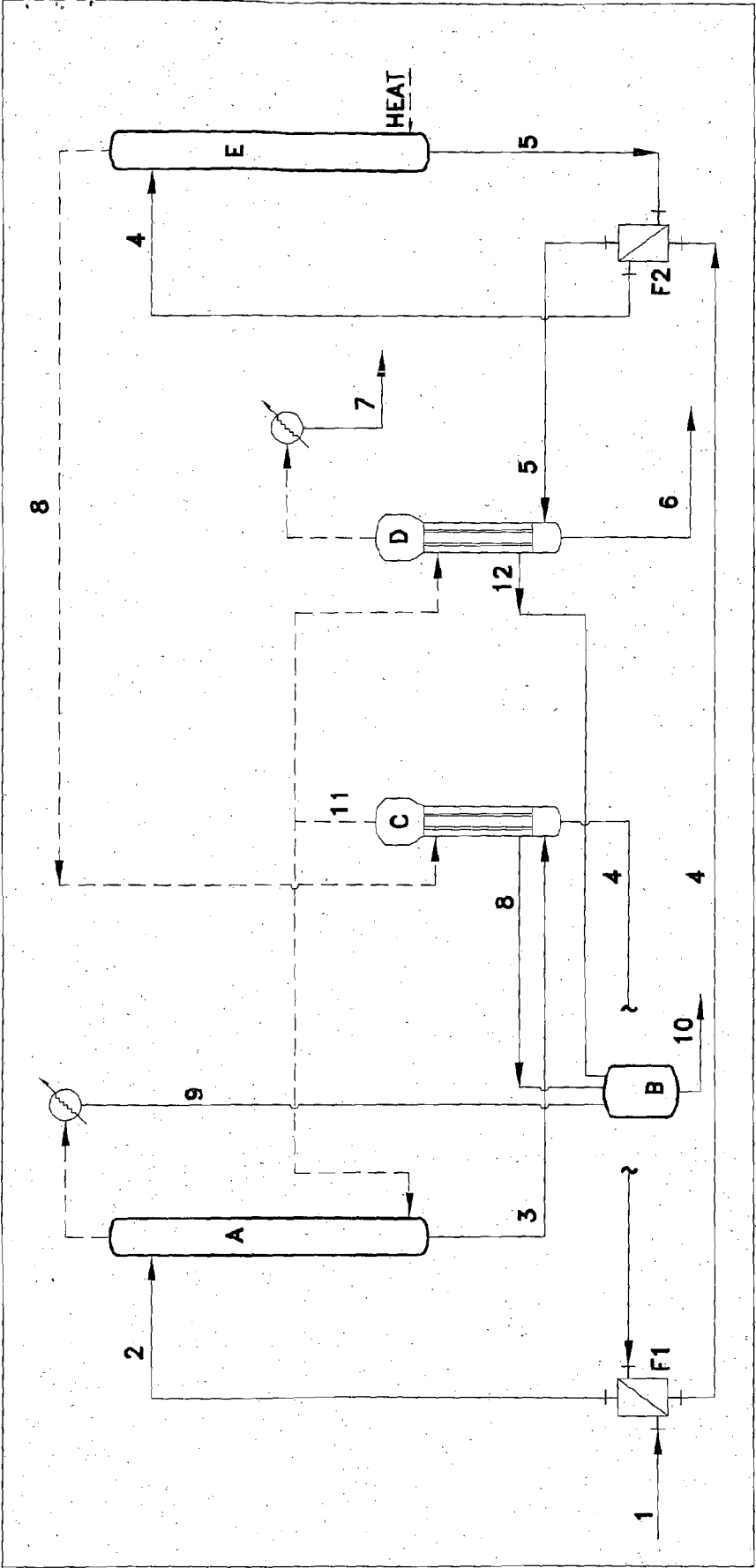


FIGURE 1

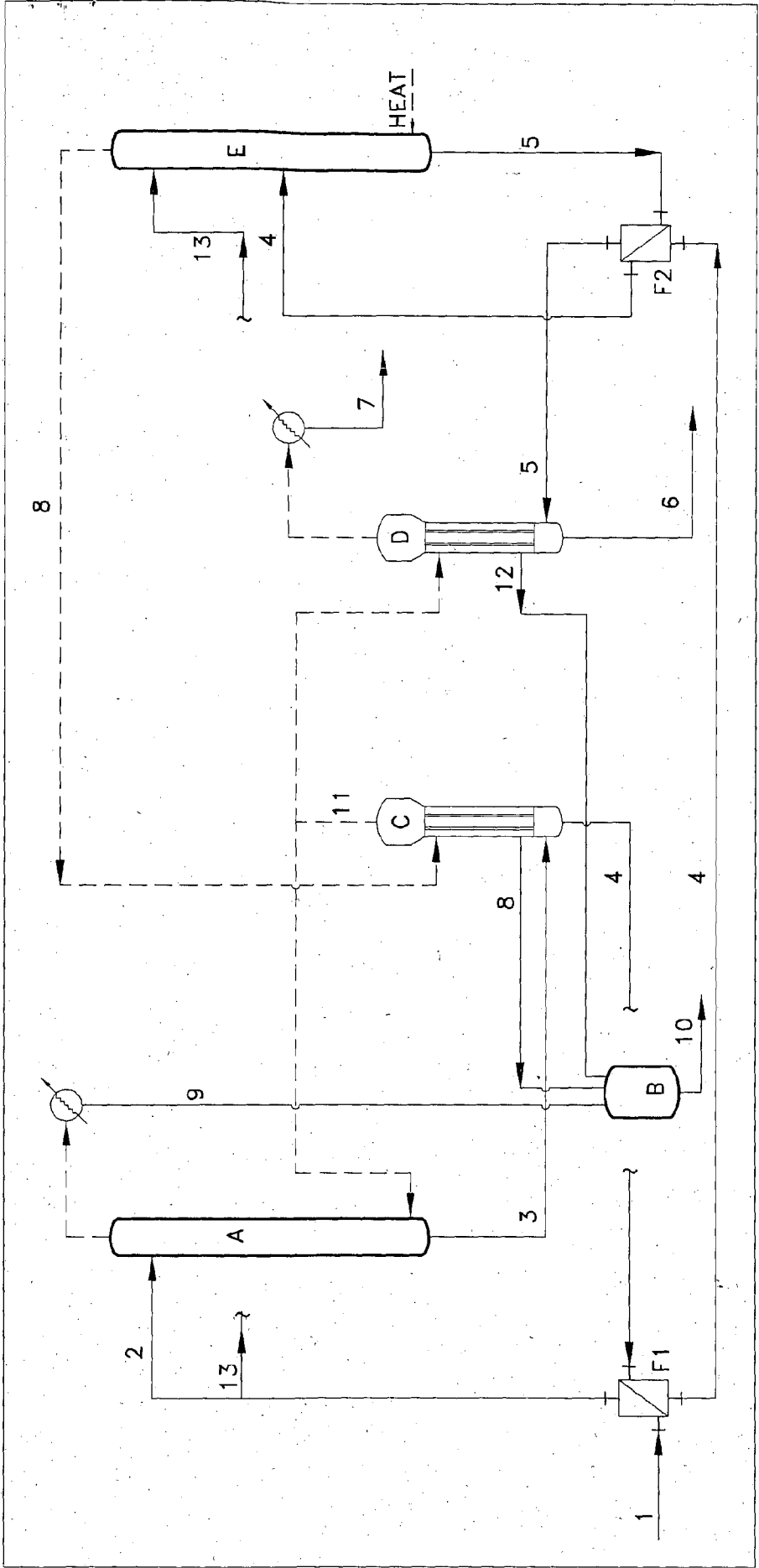


FIGURE 2