COMMONWEALTH of AUSTRALIA

PATENTS ACT 1952

**594340**

APPLICATION FOR A STANDARD PATENT

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We CANADIAN PATENTS AND DEVELOPMENT LIMITED,
of City of Ottawa,
Province of Ontario,
CANADA

$170

hereby apply for the grant of a Standard Patent for an invention entitled:

"METHOD OF SEPARATING CARBONACEOUS COMPOUNDS FROM PARTICULATE COAL CONTAINING INORGANIC SOLIDS, ~~AND APPARATUS THEREFOR~~"

which is described in the accompanying ~~provisional~~ complete specification.

Details of basic application(s):—

| Number | Convention Country | Date |
|---|---|---|
| 482,843-7 | CANADA | 30th May 1985 |

[illegible] AT [illegible]
21 MAY 1986
Melbourne

APPLICATION ACCEPTED AND AMENDMENTS ALLOWED 3-1-90

The address for service is care of DAVIES & COLLISON, Patent Attorneys, of 1 Little Collins Street, Melbourne, in the State of Victoria, Commonwealth of Australia.

Dated this 21st day of May 19 86

H. M. Pimington

(a member of the firm of DAVIES & COLLISON for and on behalf of the Applicant).

To: THE COMMISSIONER OF PATENTS

AUSTRALIAN PATENT OFFICE

Davies & Collison, Melbourne and Canberra.

CONVENTION
OR
NON-CONVENTION

FORMS 7 AND 8
(COMBINED)

# COMMONWEALTH OF AUSTRALIA

PATENTS ACT 1952-1969

## DECLARATION IN SUPPORT OF CONVENTION OR NON-CONVENTION APPLICATION FOR A PATENT OR PATENT OF ADDITION

*(The declaration shall be made by the applicant, or, if the applicant is a body corporate, by a person authorized by the body corporate to make the declaration on its behalf).*

In support of the Application made for a patent ~~patent of addition~~ for an invention entitled

Insert title of invention. METHOD OF SEPARATING CARBONACEOUS COMPOUNDS FROM PARTICULATE COAL CONTAINING INORGANIC SOLIDS AND APPARATUS THEREFOR

Insert full name(s) and address(es) of declarant(s).

~~I~~ We DOUGLAS C. CRYDERMAN and LOIS LIPKE, both of CANADIAN PATENTS AND DEVELOPMENT LIMITED/SOCIETE CANADIENNE DES BREVETS ET D'EXPLOITATION LIMITEE, a company duly incorporated under the laws of the Parliament of Canada to which the Government Companies Act applies, and having its head office in the City of Ottawa, Province of Ontario, Canada

do solemnly and sincerely declare as follows :–

Delete the words which are not applicable.

1. (a) ~~I am~~ We are ~~the applicant for the~~ patent ~~patent of addition~~

Insert full name of applicant Company

or (b) ~~I am~~ authorized by Canadian Patents and Development Limited,

the applicant..... for the patent ~~patent of addition~~ to make this declaration on ~~its~~ their behalf.

Delete the words which are not applicable.

2. (a) ~~I am~~ We are ~~the actual inventor of the invention~~

Insert full name(s) and address(es) of actual inventor(s).

or (b) C.Edward Capes of 1851 Playfair Drive, Ottawa, Ontario, Canada K1H 5R9; Richard D. Coleman of 1420 Duford Drive, Orleans, Ontario, Canada K1E 1E6; Serge Croteau of 1822 Axminster Court, Orleans,Ontario,Canada K1C 1Z4; William L.Thayer of 2057 Maywood St.Ottawa, Ontario,Canada K1G 1E8

~~I~~ are the actual inventor..s..... of the invention and the facts upon which the applicant..... ~~is~~ are entitled to make the application are as follows :–

State manner in which applicant(s) derive title from actual inventor(s).

by virtue of an Assignment dated 12 May 1986, the applicant is the assignee of the said actual inventors, in respect of the invention.



(Paragraphs 3 and 4 apply only to Convention applications).

3. The basic application..... as defined by Section 141 of the Act ~~was~~ made

Insert country and date of filing of each application on which priority is based.

in Canada on the 30th day of May 1985

by CANADIAN PATENTS AND DEVELOPMENT LIMITED

in ..... on the .....

Insert full name of applicant in each basic application.

by .....

4. The basic application..... referred to in paragraph 3 of this Declaration ~~was~~ were the first application..... made in a Convention country in respect of the invention the subject of the application.

Signature(s) of declarant(s)

Declared at Ottawa, Canada this 12th day of May 1986.

(12) PATENT ABRIDGMENT (11) Document No. AU-B-57654/86
(19) AUSTRALIAN PATENT OFFICE (10) Acceptance No. 594340

(54) Title
SEPARATING CARBONACEOUS COMPOUNDS FROM COAL

International Patent Classification(s)
(51)[4] B03D 001/02 B03D 001/16

(21) Application No. : 57654/86 (22) Application Date : 21.05.86

(30) Priority Data

(31) Number (32) Date (33) Country
482843 30.05.85 CA CANADA

(43) Publication Date : 04.12.86

(44) Publication Date of Accepted Application : 08.03.90

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(56) Prior Art Documents
EP 21778
US 3665066
AU 540510 73866/81 B03D 1/02

(57) Claim

1. A method of separating carbonaceous components from particulate coal containing inorganic solids, comprising:

(a) separating agglomerates of carbonaceous coal and oil from inorganic solids and water components of a slurry of particulate coal and water from which the agglomerates were formed;

(b) agitating the separated coal/oil agglomerates of the particulate coal in water to form an aqueous slurry of dispersed particles of the particulate coal and then intimately mixing agglomerating oil and air with the dispersed particles until robust, dense, buoyant agglomerates are formed, consisting of carbonaceous coal particles, agglomerating oil and trapped air, which accumulate at the surface of water containing newly released inorganic solids of the coal; and then

(c) separating the agglomerates from the newly released inorganic solids and the water.

COMMONWEALTH OF AUSTRALIA

PATENT ACT 1952

COMPLETE SPECIFICATION

(Original)

# 594340

FOR OFFICE USE

Class Int. Class

Application Number: 57654/86.
Lodged:

Complete Specification Lodged:
Accepted:
Published:



Priority:

Related Art:



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Name of Applicant: CANADIAN PATENTS AND DEVELOPMENT LIMITED

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1 Little Collins Street, Melbourne, 3000.

Complete Specification for the invention entitled:

"METHOD OF SEPARATING CARBONACEOUS COMPOUNDS FROM PARTICULATE COAL CONTAINING INORGANIC SOLIDS" ~~AND APPARATUS THEREFOR~~"

The following statement is a full description of this invention, including the best method of performing it known to us :-

This invention relates to a method of separating carbonaceous components from particulate coal containing inorganic solids.

It has already been proposed in United States Patent No. 3,665,066, dated May 23, 1972, " Beneficiation of Coals" , C.E. Capes et al, to add a bridging liquid to an aqueous, clay containing slurry of coal fines, then agitate the resultant mixture to form coal agglomerates dispersed in a slurry of the residual clay and ash impurities, and then separate the coal agglomerates by skimming them through an overflow spout in a float-sink tank. The separation of the coal agglomerates may be assisted by introducing a multitude of air bubbles at the bottom of the float sink tank.

While the separation process taught by Capes et al has proved to be useful, it would be desirable to provide a process wherein:

i) there is no need for a float sink tank,

ii) the agglomerates themselves are rendered more buoyant and are thus rendered much more easily to separate from the residue slurry, and

iii) where agglomerates have already been formed, as taught by Capes et al, they are broken down and reformed to release ash trapped therein and render them

more buoyant for ease of separation from a slurry of the ash.

According to the present invention there is provided a method of separating carbonaceous components from particulate coal containing inorganic solids, comprising:

(a) separating agglomerates of carbonaceous coal and oil from inorganic solids and water components of a slurry of particulate coal and water from which the agglomerates were formed;

(b) agitating the separated coal/oil agglomerates of the particulate coal in water to form an aqueous slurry of dispersed particles of the particulate coal and then intimately mixing agglomerating oil and air with the dispersed particles until robust, dense, buoyant agglomerates are formed, consisting of carbonaceous coal particles, agglomerating oil and trapped air, which accumulate at the surface of water containing newly released inorganic solids of the coal; and then

(c) separating the agglomerates from the newly released inorganic solids and the water.

The robust, dense buoyant agglomerates may be removed from the slurry water by known techniques such as skimming or screen separation.

Preferably, at least 0.3 weight % of agglomerating oil is added to the aqueous slurry based on the weight of the solids content of the slurry.

Water may be removed from the agglomerates.

A conditioning agent for increasing the oil wettability of the coal and/or a frothing agent may be added to the slurry.

The previously formed inorganic solids reduced coal/oil agglomerates may be formed by such processes as the known high shear and low shear agglomerating processes. By using previously formed, inorganic solids reduced, coal/oil agglomerates, the carbonaceous coal particles have been previously conditioned with oil and as a consequence are thoroughly mixed with the oil and air during the formation of the slurry and then the formation of agglomerates from the slurry. This will typically result in the release of more inorganics and will result in the formation of surprisingly robust, dense, buoyant agglomerates. This is believed to occur because less inorganic substances are present in the agglomerates and more intimate contact between oiled carbonaceous particles and trapped air in the agglomerates is achieved. The attraction between carbonaceous particles may be so great that even if the inorganic substances remain in the water used to preform the agglomerates and this water is used to form the slurry, these inorganic substances together with newly released inorganic substances will remain in the water when the robust, dense, buoyant agglomerates are formed.

In the accompanying drawings which illustrate, by way of example, embodiments of the present invention,

Figure 1 is a schematic side view of an apparatus for separating carbonaceous components from particulate coal containing inorganic solids;

Figure 2 is a similar view to Figure 1, but of a different apparatus;

Figure 3 is also a similar view to Figure 1, but of yet another, different apparatus; and

Figure 4 is a schematic view of a more complex apparatus for separating carbonaceous components from particulate coal containing inorganic solids.

Referring now to Figure 1 there is shown a beaker 1 and a stirrer, generally designated 2. The stirrer comprises a glass tube 4, a porous, sintered glass tip 5 fused to the lower end of the glass tube 4, and a flexible tube 6 for connection to a pressurized air supply (not shown).

When the apparatus shown in Figure 1 was used to verify the present invention, an agueous slurry 8 of particulate, inorganic solids containing, coal was poured into the beaker 1 together with agglomerating oil.

Pressurized air was fed along the tube 6 and emitted from the tip 5 as fine bubbles which rose up through the slurry 8. The tube was rapidly stirred in the direction X and agglomerates of the carbonaceous portion of the coal and oil were formed with air trapped in them. The trapped air gave the agglomerates sufficient buoyancy for them to rise and collect at the surface of the water where they could easily be removed. Ash residue from the coal was found to settle at the bottom of the water.

Referring now to Figure 2, there is shown a conventional blender mixing cup 10 and base 12 containing a motor drive for an impeller shaft 14 rotatably sealed to and extending through the base of, the cup 10. An impeller generally designated 16 has blades 17 to 20 shaped for drawing air to form an air vortex in, and aerating, and agitate, a slurry in the cup 10.

When the apparatus shown in Figure 2 was used to verify the present invention, an aqueous slurry 22 of particulate, inorganic solids containing, coal was poured into the cup 10. The impeller 16 was then rapidly rotated in the direction of arrow Y to form an air vortex 24 in, and aerate, and agitate, the slurry 22. Agglomerates of the carbonaceous portion of the coal and oil were formed with air trapped in them. The

trapped air gave the agglomerates sufficient buoyancy for them to rise and collect at the surface of the water where they could easily be removed. Ash residue from the coal was found to collect in a lower portion of the water.

Referring now to Figure 3, there is shown a container 26 and an impeller assembly generally designated 28. The impeller assembly 28 comprises an impeller 30, an impeller shaft 32, with the impeller 30 mounted on a lower end thereof, an air conduit 34 coaxial with and spaced outwardly from the shaft 32 and sealed at the upper end to the shaft 32 for the shaft 32 to rotate therein, air inlet means 36 to an upper end portion of the conduit 34, and a cylindrical casing 38 around the impeller 30, the casing 38 having an upper annular-shaped agglomerate inlet 40 extending around the exterior of lower, air outlet end of the conduit 34 and a plurality of arcuate, agglomerate outlets, such as outlets 42 and 44, around the casing and spaced radially outwardly from the impeller 30 for agglomerates formed by the impeller 30 with air trapped in them to be ejected centrifugally therethrough.

When the apparatus shown in Figure 1 was used to verify the present invention, previously formed coal/oil agglomerates and water were poured into the container 26 together with agglomerating oil. Pressurized air was

fed to the inlet 36 from a source (not shown) and the shaft 32 was rotated in the direction of arrow Y. The previously formed coal/oil agglomerates and water were drawn by the impeller 30 into inlet 40 where the agglomerates were broken down and carbonaceous portions of the coal and oil reformed as newly formed agglomerates with air trapped in them from the conduit 28. Any residual ash that was present in the previously formed coal/oil agglomerates was left in the water. The newly formed agglomerates collected at the top of the water while the ash residue collected at the bottom of the container 26.

The apparatus shown in Figure 3 can also be used by pouring an aqueous slurry of the particulate, inorganic solids containing, coal in the container 26.

In Figure 4 there is shown a tank 46 having outlet pipes 48 and 50 and a return pipe 52. The pipes 48 and 50 are connected to an inlet side of a centrifugal pump 54. Valves 56 and 58 are provided in the outlet pipes. The return pipe 52 is connected to the outlet from the pump 54 and contains a valve 60. An air pipe 62 is also connected to the inlet side of the pump 54. The tank 46 has an agglomerate overflow weir 64 for delivering agglomerates to a screened, dewatering vacuum filter 66 which is connected by a pipe 68 to a wet vacuum system 70. An agglomerate storage vessel 72 is provided.

In operation, previously formed agglomerates, which were produced using the conventional high shear and then low shear mixers, were poured into the tank 46 together with water and formed into a slurry. The valves 56 and 60 were opened, the pump 54 was started, and air fed to the pump along pipe 62, so that the slurry was drawn along the pipe 48 and returned aerated along the pipe 52. The aeration caused dense, wet agglomerates to form of carbonaceous components of the coal and oil and containing trapped air, which collected at the surface of the slurry and could easily be skimmed over the weir 64. Fresh water was added periodically.

Batches of the dense, wet agglomerates were spread, one after another, over the screen of the filter 66 and the vacuum system 70 was operated to dewater the agglomerates. After each batch was dried it was transferred to vessel 72 for storage.

When the formation of agglomerates diminished in the tank 46 the valve 58 was opened to pump water containing residual ash from the tank 46 along pipe 74 to a water clarifier (not shown).

The following tables give the results of tests that were carried out to verify the present invention.

Table I gives the results of tests carried out with a coal which does not easily respond to oil

agglomeration. In these tests a conditioner and/or a frothing agent were found to be desirable for good recovery of the coal combustibles ( which were essentially the carbonaceous components). The results of the tests given in Table I are for coal agglomerates which had been previously formed with trapped air, using the apparatus shown in Figure 2, but which were broken down with the original water and ash and then re-formed into agglomerates using the apparatus shown in Figure 1, and then recovered.

TABLE I

BITUMINOUS COAL TAILINGS SLURRY FROM WESTERN CANADA CONTAINING 45% WEIGHT ASH

AGGLOMERATE RECOVERY PROCESS USING 3.5% WEIGHT NO. 4 FUEL OIL, WITH OXIDIZED COAL CONDITIONER AND/OR FROTHING AGENT EACH ADDED IN AMOUNTS IN THE RANGE 0.03 WEIGHT % TO 0.15 WEIGHT % BASED ON THE TOTAL SOLIDS CONTENT OF THE SLURRY

| CONDITION | AGGLOMERATES | |
|---|---|---|
| | Weight % Ash | Combustibles Recovered from Coal Tailings Slurry in weight % of that originally present |
| No. 4 oil only | 13.4 | 24 |
| Conditioner + Frothing Agent | | Negligible |
| No. 4 oil + Frothing Agent | 13 | Approaching 80 |
| No. 4 oil + Conditioner | 13 | 85 Maximum |
| No. 4 oil + Conditioner + Frothing Agent | 15 | Approaching 90 |

In Tables II and III, agglomerates previously formed by the known high shear and low shear coal/oil agglomerating process of an easily oil agglomerated coal still present in the water and inorganics which were originally present in the slurry from which the agglomerates were formed, were broken down and reformed as agglomerates using the apparatus shown in Figure 3.

In Tables II and III, d.b. is the weight of solids present in the feed, MM is the mineral matter, and Pulp is the d.b. as a weight % of the total weight of the feed.

TABLE II

FEED No. 1
Agglomerated with 1.0% #4 oil
at 30% solids and diluted
to 10% solids in low shear mixer

| Batch | Sample | Wt % Oil | | Product | | | | Tails | | | | Wt % Ash in Feed | Wt % S in Feed MM Free | Pulp Density |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | d.b. feed | d.b. prod | Wt % ash d.b. | Wt % S d.b. MM Free | Mass Yield | Comb. Rec. | Wt % ash d.b. | Wt % Ash Rej. | Wt % S d.b. MM Free | S Rej. | | | |
| 3 | 0-A | 1.00 | 1.57 | 13.13 | 3.74 | 63.59 | 89.35 | 81.91 | 78.13 | 27.31 | 46.54 | 38.17 | 6.25 | 10.65 |
| 3 | 30-A | 1.00 | 1.58 | 14.62 | 3.90 | 63.25 | 88.82 | 81.50 | 76.41 | 28.16 | 47.61 | 39.20 | 6.61 | 10.63 |
| 3 | 1-A | 1.00 | 1.56 | 13.47 | 3.62 | 63.96 | 89.86 | 82.67 | 77.57 | 29.20 | 47.67 | 38.41 | 6.21 | 10.34 |
| 3 | 2-A | 1.00 | 1.58 | 13.67 | 3.98 | 63.31 | 89.14 | 81.85 | 77.63 | 27.27 | 45.47 | 38.68 | 6.51 | 10.52 |
| 3 | 5-A | 1.00 | 1.55 | 14.46 | 3.32 | 64.68 | 91.24 | 84.96 | 76.24 | 34.18 | 49.71 | 39.36 | 6.02 | 10.33 |
| 3 | 10-A | 1.00 | 1.54 | 14.43 | 3.53 | 64.99 | 92.35 | 86.83 | 76.42 | 37.36 | 46.74 | 39.77 | 6.[illegible] | 10.60 |

TABLE III

Feed No. 2
Agglomerated with 1.5% #4 oil at 30% solids and diluted to 10% solids in low shear mixer

| Batch | Sample | Wt % Oil | Product | | | | Tails | | | | Wt % Ash in Feed | Wt % S in Feed MM Free | Pulp Density |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | d.b. Feed | Wt % ash d.b. | Wt % S d.b. MM Free | Mass Yield | Comb. Rec. | Wt % ash d.b. | Wt % Ash Rej. | % S d.b. MM Free | S Rej. | | | |
| 6 | 0-A | 1.50 | 16.09 | 3.53 | 60.36 | 88.27 | 83.02 | 77.21 | 29.80 | 52.88 | 42.62 | 6.61 | 11.01 |
| 6 | 30-A | 1.50 | 14.36 | 3.54 | 58.05 | 86.21 | 81.04 | 80.31 | 25.11 | 53.17 | 42.34 | 6.51 | 10.64 |
| 6 | 1-A | 1.50 | 15.82 | 3.64 | 54.64 | 74.43 | 65.15 | 77.37 | 11.71 | 52.53 | 38.19 | 5.70 | 12.07 |
| 6 | 2-A | 1.50 | 16.77 | 3.50 | 60.34 | 87.69 | 82.23 | 76.32 | 27.24 | 52.23 | 42.73 | 6.42 | 10.54 |
| 6 | 5-A | 1.50 | 15.52 | 3.68 | 57.44 | 86.44 | 82.12 | 79.68 | 29.59 | 55.76 | 43.86 | 7.19 | 11.46 |
| 6 | 10-A | 1.50 | 18.23 | 4.79 | 63.34 | 89.60 | 83.60 | 72.63 | 28.84 | 41.12 | 42.19 | 7.29 | 11.21 |

The tests showed that:

i) frothing agents such as, for example, those marketed under the trademark Aerofroth 76, by Cyanamid Canada Inc., Willowdale, Ontario, Canada, and methyl isobutyl carbinol were useful additions to the slurry for nucleating air bubbles, and

ii) where clay is present, or where the coal is difficult to wet with oil (e.g. oxidized coal) a conditioning agent for increasing the oil wetability of the coal, such as, for example, the surfactant marketed under the trademark Accoal-4433, by Cyanamid Canada Inc., Willowdale, Canada were useful additions to the slurry.

The present invention provides a useful starting material for producing the water continuous phase fuel described and claimed in United States Patent Application No. 656,675, filed October 1, 1984, " Aqueous Phase Continuous, Coal Fuel Slurry and a Method of its Production", Capes et al.

THE CLAIMS DEFINING THE INVENTION ARE AS FOLLOWS:-

1. A method of separating carbonaceous components from particulate coal containing inorganic solids, comprising:

(a) separating agglomerates of carbonaceous coal and oil from inorganic solids and water components of a slurry of particulate coal and water from which the agglomerates were formed;

(b) agitating the separated coal/oil agglomerates of the particulate coal in water to form an aqueous slurry of dispersed particles of the particulate coal and then intimately mixing agglomerating oil and air with the dispersed particles until robust, dense, buoyant agglomerates are formed, consisting of carbonaceous coal particles, agglomerating oil and trapped air, which accumulate at the surface of water containing newly released inorganic solids of the coal; and then

(c) separating the agglomerates from the newly released inorganic solids and the water.

2. A method according to claim 1, wherein at least 0.3 weight % of agglomerating oil is added to the aqueous slurry based on the weight of the solids content of the slurry.

3. A method according to claim 1 or claim 2, wherein water is removed from the agglomerates.

4. A method according to any preceding claim, wherein a frothing agent is added to the aqueous slurry.

5. A method according to any preceding claim wherein a conditioning agent for increasing the oil wettability of the coal is added to the slurry.

6. A method according to claim 1 for separating carbonaceous compounds from particulate coal containing inorganic solids substantially as hereinbefore described with reference to the drawings.

DATED this 20th day of December 1989.

CANADIAN PATENTS AND DEVELOPMENT LIMITED
By its Patent Attorneys
DAVIES & COLLISON

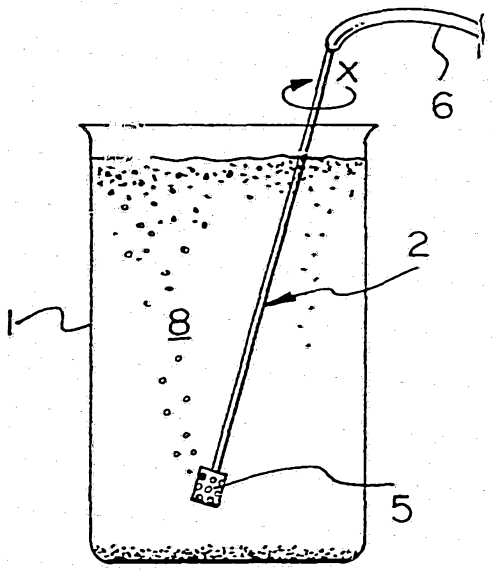


FIG. 1

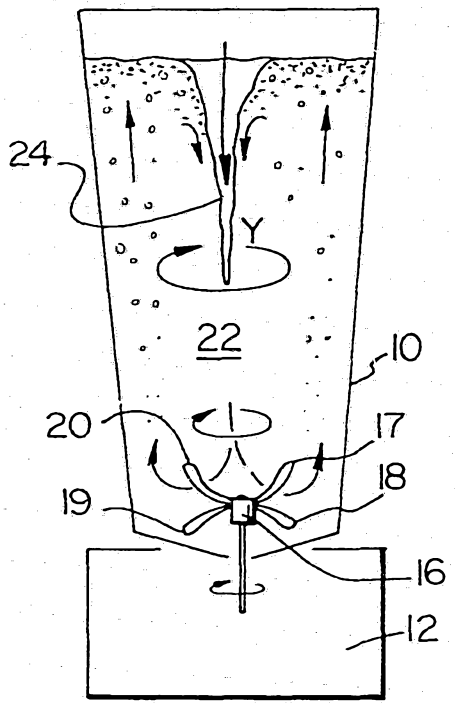


FIG. 2

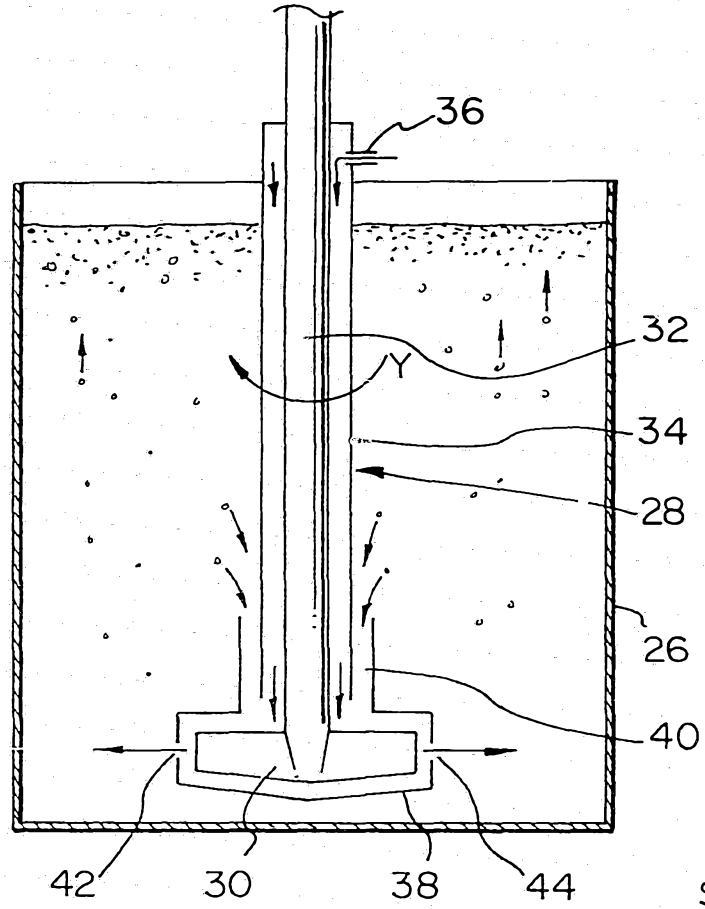


FIG. 3

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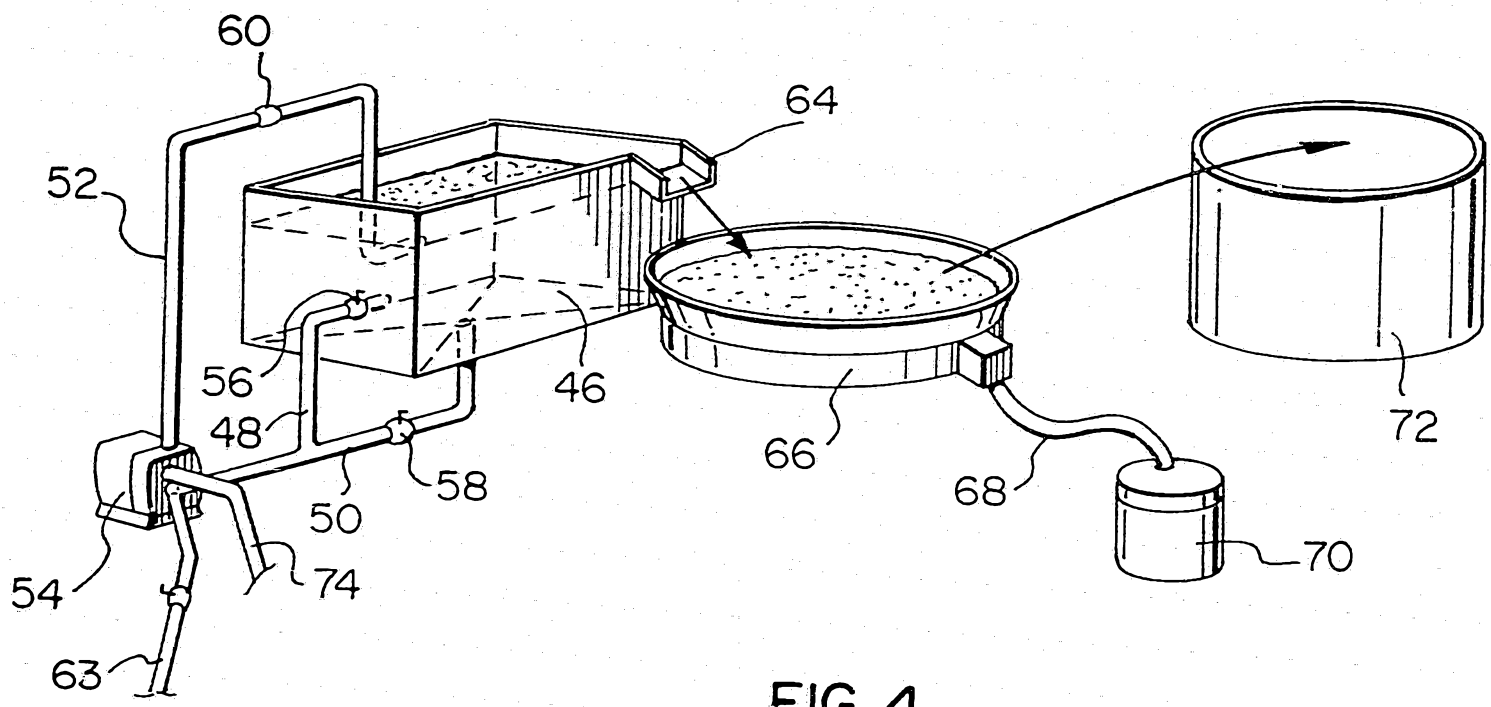


FIG. 4