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(57) Abstract

In accordance with the present invention, there is provided an asphalt composition which incorporates asphalt in admixture with synthetic or natural rubber and a vulcanizing agent. There is also provided a process for preparing the above asphalt composition. Preferably, the rubber is obtained from waste tires or tubes.

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ASPHALT COMPOSITION AND PROCESS FOR OBTAINING SAME

TECHNICAL FIELD

The invention relates to an asphalt (or bitumen) composition, and its process of preparation.

BACKGROUND OF THE INVENTION:

The elimination of used tires is becoming an increasingly difficult problem throughout the world. As an example of the importance of the problem, over 200,000 tons of waste tires and tubes are produced each year in Canada. In the United States of America, that amount is close to 4,000,000 tons.

Although it is very attractive to consider the possibility of using waste rubber products such as tires or tubes in asphalt (or bitumen) compositions, there are a number of significant obstacles related to such use. Scrap tires contain different types of vulcanized rubber, for example polyisoprene, polybutadiene, styrene-butadiene, butyl, ethylene-propylene-diene, as well as fillers, plasticizers, surfactants and the like. Sometimes, tires contain up to 31 different elastomers. Ground rubber or crumb rubber does not disintegrate readily, because of the cross-linking of the polymeric material that takes place by the vulcanization process with sulfur. The additional chemical bonds created during this vulcanization render impossible the complete dissolution of the polymer in hot bitumen, thus causing problems of stability and storage of the material. Furthermore, such mixtures are not homogeneous, and form two phases of different density which are partially dispersed one in the other. The system tends to decant and causes the upper layers, which have a lower density, to be constituted of polymer, while the lower layers, which have a higher density, are constituted of bituminous material.

At high temperature, conventional asphalt compositions, that is asphalt cement for use in pavement which meet ASTM-D-946-74 or ASTM-D-3381-83 specifications, become fluid and even sticky, causing aggregate deformation or rutting, while at lower temperatures, they become fragile and crack easily. Toughness and durability of asphalt compositions are directly proportional to the resistance of the asphalt to deformation under the effect of stress.

DE 38 19 931 discloses polymer-modified bitumen compositions comprising bitumen in admixture with an olefin, the latter having been oxidized before being mixed with bitumen. These compositions are alleged to be binders in

hot-mix asphalt such as pavements. These compositions, as useful as they could be, are however extremely expensive, because of the use of virgin olefins, that is, brand new synthetic olefins which have never been used before.

CA 1,109,191; CA 1,209,295; CA 1,137,242 and CA 1,137,243 (corresponding to US 4,145,322; US 4,554,313; US 4,567,222; US 4,242,246 and US 4,330,449) disclose processes of preparation of asphalt compositions which comprise mixing vulcanized virgin olefins with conventional bitumen. The vulcanization is performed with sulfur or polymeric sulfur derivatives. As explained above, such compositions are extremely costly because of the use of virgin olefins.

US 4,992,492 discloses a bitumen and reclaimed rubber powder binder for surfacing roadways. The material and the process for preparing it are however limited by the size of the particles of reclaimed rubber. If the size of the particles is too important, the resulting mixture, or binder, is unstable, non homogeneous, and cannot be stored. It is also obvious that the smaller the particles of reclaimed rubber are, the higher their cost.

US 4,437,896 is concerned with synthetic bitumen compositions comprising gilsonite, tall oil and oil pitch. Again the size of the reclaimed rubber particles that can be used in the process of this patent cannot exceed 850 microns, which constitutes an important drawback.

US 4,609,696 teaches asphalt emulsion, which may contain reclaimed rubber. The particles involved in the process are also limited to a size of 850 microns, and preferably 300 microns.

It would therefore be highly desirable to obtain an asphalt composition having good impact resistance at low temperature and excellent rheological and mechanical properties. Improving these properties over a range of temperatures provides a superior load bearing capacity of pavement, and a better ability to resist to ground movements or other stresses. Waste rubber such as waste tires and tubes would preferably be used in the composition. Preferably, such asphalt composition would be obtained through a versatile and inexpensive process.

In order to further reduce the costs of such asphalt composition, it would be necessary that it be prepared with rubber particles of the greatest possible size.

SUMMARY OF THE INVENTION:

In accordance with the present invention, there is provided an asphalt composition suitable for pavement, shingle and the like, which incorporates synthetic or natural rubber cross-linked with a vulcanizing agent. More specifically, the present invention is concerned with an asphalt composition comprising:

- from 60 to 99 % by weight of asphalt (or bitumen);
- from 1 to 40 % by weight of synthetic or natural rubber dissolved in the asphalt; and
- from 0.01 to 3 % by weight of a vulcanizing agent.

The present invention is also concerned with a process for preparing the above asphalt composition, which comprises the steps of:

a) mixing asphalt with synthetic or natural rubber at a high temperature in order to have the rubber particles in suspension;

b) injecting air in the mixture of step a) under high pressure;

c) homogenizing the mixture with a dispersing device;

d) stabilizing the mixture by incorporating the vulcanizing agent therein; and

c) recovering the asphalt composition.

In a preferred embodiment, the rubber consists in waste tires or tubes.

In a second aspect of the process of the present invention, the mixture of step a) remains in a reactor for a period of time varying from 1 to 15 hours wherein air is injected, if the rubber used is waste tires or tubes. A second reactor, preferably adjacent to the first reactor, may be incorporated in the system when a longer period of devulcanization of the waste tires or tubes is required.

IN THE DRAWINGS

Figure 1 illustrates a first embodiment of a system suitable for carrying out the process of the present invention;

Figure 2 illustrates a second embodiment of a system suitable for carrying out the process of the present invention; and

Figure 3 illustrates a third embodiment of a system suitable for the carrying out the process of the present invention.

DETAILED DESCRIPTION OF THE INVENTION:

As indicated above, the present invention provides an asphalt composition which incorporates asphalt in admixture with synthetic or natural rubber dissolved therein, and a vulcanizing agent. Waste rubber obtained from old tires is the preferred starting material for obvious economic reasons. In view of the generalized concern towards the environment, the present invention represents an important step forward, since it allows the recuperation of waste rubber originating from old tires or tubes. Furthermore, the resulting asphalt composition is simple to prepare, with the costs of starting materials and experimental procedures being reduced significantly.

It is common knowledge that the rubber employed in the manufacture of tires and tubes is vulcanized, that is, sulfur bonds have been introduced in the polymeric chain, with the result that the physical properties of the material thus treated are improved while its elasticity is maintained. To be suitable for the purposes of the present invention, such waste rubber is treated to be devulcanized by dehydrogenation, preferably with air injected under pressure and at high temperature. To the resulting devulcanized rubber is then added a vulcanizing agent. Examples of suitable vulcanizing agents include elemental selenium or organo-selenium such as AC-8111 or UN-2658, manufactured and sold by Anachemia, or Refined Selenium Glass Garde manufactured and sold by Noranda Minerals Inc; elemental sulfur or organo-sulfur, for example those described in US 4,554,313.

Vulcanizing accelerators may also be included during the process of the present invention. These accelerators advantageously permit to reduce the time of vulcanization and increasing the degree of cross-linking, without affecting the

properties of the final product. They can be incorporated in an amount varying from 0.01 to 3% by weight with respect to the amount of rubber or waste rubber present in the composition. Examples of such accelerators include thiazol or thiuram derivatives such as tetramethyldiuram disulfide, or a conventional mixture of zinc oxide and stearic acid. The composition may contain more than one accelerator. A preferred mixture of accelerators is tetramethyldiuram disulfide:zinc oxide:stearic acid in a ratio 2:2:1.

Referring to the drawings, Figure 1 shows a scheme of the first embodiment, referred to as system **10**, suitable for the preparation of the asphalt composition of the present invention. In detail, rubber particles are brought into mixer **12** from dispenser **14** through conveyor **16**, while asphalt (bitumen) is brought into mixer **12** from tank **15** through pipe **18**. The temperature of the mixture in mixer **12** is maintained at 160-200°C and the mixture remains therein under agitation until the rubber particles are uniformly suspended. The resulting blend is then forwarded through pipe **22** into exchanger **20**, wherein the temperature is further raised to about 230°C. The blend thus heated is placed in a reactor **24** for a period of time varying from about 1 to 12 hours, depending on the intended use of the asphalt composition, during which air is injected in the reactor, preferably from the bottom, at a pressure of from about 10 to about 50 psi (pounds per square inch). It is to be noted that the pressure could be higher depending on the height of the reactor. The gas produced in reactor **24** is preferably purified by air filter or purifier **26** before being rejected in the atmosphere.

The blend is subsequently forwarded into dispersing device **28** through pipe **30**, to homogenize the blend. Pressurized air is injected through a hole in pipe **30** to ensure completeness of the dehydrogenation reaction. Dispersing device **28** consists in a pair of toothed discs turning at 3600 RPM, which permit the homogenization, as well as the acceleration of the dehydrogenation reaction. Additives are preferably added in the blend prior to the homogenization. It is imperative that the liquid composition be homogenized to eliminate storage problems. The vulcanizing agent as defined above is then added into the homogenized composition, which subsequently proceeds through static mixer **32** before being recovered and stored in container **34**. The storage temperature in container **34** is preferably about 150-175°C. The system **10** illustrated in Figure 1

and the corresponding process provides an asphalt composition suitable for pavements, shingles, or any other industrial or domestic uses of asphalt. Some minor experimental adjustments may however be necessary, such as the temperature, the pressure, allowed time in the reactor and the like, depending on the intended use of the asphalt composition and the vulcanizing agent used. All these adjustments will become apparent to those skilled in the art.

The system **11** illustrated in Figure 2 is similar to the one shown in Figure 1, with the exception that reactor **24** has been removed. This system is suitable for preparing asphalt composition to be utilized for pavements since such compositions require a shorter reaction time, because the asphalt needs to be softer.

The system **17** illustrated in Figure 3 is also similar to system **10** of Figure 1, except that a second reactor **36** has been incorporated in the system. This system is preferred for the preparation of shingle or roofing products, which requires much harder and tougher asphalt. Though more expensive than the preceding systems, system **17** permits a satisfactory volume of asphalt to be produced in a given period of time.

The key steps in the process of the present invention are the injection of air in the blend followed by homogenization in the dispersing device, particularly when the rubber particles are obtained from waste tires as described above. In this specific situation, if air is not injected, it is not possible to obtain a homogeneous composition after the passage of the blend into dispersing device **28**, which results in the production of material of poor quality and stability.

Another critical point to consider is the fact that the rubber composition of waste tires or tubes is different, sometimes significantly, from one manufacturer to another. Accordingly, many samples were tested to verify their processability and the properties of the asphalt composition containing same. The results have shown that any kind of used tires can be incorporated in the asphalt composition of the present invention with satisfactory results.

Another important feature of the present invention, is the size of the particles of waste rubber which can be used. Typically, the particles can be of a size

of up to ½ inch thick. Such particles are about ten times bigger than those presently employed.

It will become obvious to anyone skilled in the art that non-vulcanized waste rubber can also be used in the preparation of the asphalt composition of the present invention.

The composition of the asphalt containing the waste rubber products will depend on the particular requirements in accordance with its intended use. The range of each component is as follows:

- from 60 to 99 % by weight of asphalt, preferably from 80 to 95 %;
- from 1 to 40 % by weight of rubber, preferably from 5 to 20 %; and
- from 0.01 to 3 % by weight of vulcanizing agent, with a preferred concentration of from 0.1 to 0.3 %.

As an example of synthetic rubber capable of being used instead of devulcanized waste rubber, there may be mentioned styrene-butadiene-styrene rubber (SBS), styrene-butadiene-rubber (SBR), polyethylene, polyisoprene, polybutylene, polychloroprene (neoprene), nitrile rubber (acrylonitrile butadiene), butyl rubber (copolymer of isobutylene and isoprene) polyacrylonitrile and the like.

Since the price of waste tires is relatively low, particularly for large particles, the costs related to the preparation of the asphalt composition of the present invention are drastically reduced when compared to the costs of preparation of conventional asphalt compositions. Accordingly, it becomes obvious that the use of waste tires or tubes represents the most preferred embodiment of the present invention. In addition to the reduced costs, the present invention permits the reclaiming of used tires and tubes which would otherwise pollute the environment in tire dumps.

The consistency of the asphalt can be modified with a so-called petroleum fraction, which can be described as a light or medium cycle oil obtained from the distillation of oil. Such product, for example L.C.O. (Light Cycle Oil) manufactured and sold by Shell Oil, is readily available from any company involved in oil refining, and is incorporated in the mixture together with the rubber particles.

The concentration of this petroleum fraction in the asphalt composition varies from 2 to 20 % by weight of the final composition, depending on the mechanical properties required.

Preferably, the dehydrogenation is carried out at a temperature of about 200 - 260°C. Such dehydrogenation process is well known in the art, and can be carried out in any of the systems illustrated in Figures 1-3, or any other conventional systems for industrial dehydrogenation purposes. After the addition of the vulcanizing agent, the mixture is agitated for a period of time varying from 30 minutes to 3 hours at a temperature of 160 -180°C depending on the starting materials and the properties required for the asphalt composition.

Polymeric additives such as styrene-butadiene-styrene (SBS), styrene-butadiene-rubber (SBR) and the like can be added in a concentration of from 0 to 5 % by weight of the final asphalt composition. These additives improve the elongation properties and elasticity of the asphalt. Again, the concentration of these additives will depend on the desired specifications of the asphalt composition.

The process of the present invention is more efficient and especially more economical than the existing conventional processes. The time of reaction is greatly reduced, which results in a very limited degradation, if any, of the materials present in the composition. Furthermore, the process of the present invention does not produce liquid or solid wastes, and possesses an enhanced capacity of production.

The rheological and mechanical properties of the asphalt composition of the present invention referred to in the examples, and more particularly in Tables 1 and 2, are as follows:

- PVN: penetration and viscosity number (CANCGSB-2) 16-3-M-90;
- the penetration is measured by the standardized procedure NQ-2300-270 or ASTM-D-5;
- the softening point is measured by the ring and ball method ASTM-D-36;
- the viscosity is measured using NQ-2300-600 or ASTM-D-21-70;
- the Frass point (fragility point) is measured using ZP80-53; and

- the storage stability is measured using DGA-S-5610-4;

The elasticity, resilience and ductility tests were performed under the current standard methods fixed by the ASTM Committee.

The following Examples are provided to illustrate the present invention rather than limit its scope. It should be noted that the expression "crumb rubber" cited in the Examples, refers to rubber obtained from waste tires or tubes.

EXAMPLE 1

A mixture of 8.37 kg of Asphalt 150/200 pen, 0.80 kg of petroleum fraction and 0.80 kg of crumb rubber is heated at 250°C and stirred for 2 hours under pressurized air stream in a dehydrogenation tower. Subsequently, 0.03 kg of selenium are added, and the resulting mixture is homogenized in a dispersing device for 2 hours. The physical properties of the resulting asphalt composition are illustrated in Table 1.

EXAMPLE 2

Following the procedure of Example 1, but reducing the concentration of Asphalt 150/200 pen to 8.27 kg, and incorporating 0.15 kg of styrene-butadiene-styrene (SBS), another asphalt composition was obtained. The physical properties of this composition are also illustrated in Table 1.

TABLE 1

	Composition of Example 1	Composition of Example 2
Penetration at 25°C	93 1/10 mm	85
Viscosity	650 cst	900 cst
P.V.N.	0.43	0.80
Softening point	52°C	60°C
Penetration index	1.0	2.0
Frass point	-20°C	-25°C
Storage stability	0.7°C	0.7°C

The results provided in Table 2 further illustrate the advantages of the asphalt composition of the present invention as compared to the presently existing conventional asphalt composition.

TABLE 2

	Conventional 80 - 100	Composition of Example 1	Composition of Example 2
Penetration at 25°C 100 g, 5 s, 0.1 mm	80 - 100	93	85
Penetration at 4°C 200g, 60 sec, 0.1 mm	43.5	62	64
% Penetration 4°C/25°C	47.8	65	75
Flash point (°C)	> 230	> 230	> 230
Viscosity at 135°C (mm^2/s)	260 (B) 185 (C)	630	900
Softening point (°C)	40	52	60
Ductility at 4°C 5 cm/min (cm)	6	10	10
Solubility (%weight) in trichloroethylene	99.0	95.0	95.0
P.V.N.	- 0.65	+ 0.43	+ 0.80
Elasticity at 10°C	—	40 %	60 %
Ratio strength/ ductility	—	0.20	0.40
Resilience	—	11	18

EXAMPLE 3

40 tons of an asphalt composition of the present invention were prepared in the following manner: A mixture comprising 89.9% by weight of asphalt 157 pen and 10% by weight of crumb rubber (size of the particles = 20 mesh) are blended for 0.5 hour at 230°C. Air is then injected at a pressure 50 psi at a 75 cubic feet per minute (CFM), and the blend is then homogenized in the dispersing device, which has a capacity of 40 tons/hour, for 2 hours. Subsequently, 0.1% of elemental selenium is introduced in the homogenized product, and further agitated for 2 to 3 hours. The asphalt composition recovered can be stored for an extended period of time without suffering from any instability problems. The test results of this composition are illustrated in Table 3.

TABLE 3

PROPERTIES (TYPICAL)	BEFORE TFOT	AFTER TFOT
PENETRATION 25°C / 100g / 5sec 4°C / 100g / 5sec 4°C / 200g / 60sec	 83.5 dmm 16.7 dmm 51.8 dmm	 56.8 13.8 42.8
VISCOSITY (135°C)	636 mm²/s	965
SOFTENING POINT	51.5°C	56°C
DUCTILITY (4°C)	7.5cm	4.0cm
PEN 4°C/PEN 25°C	62%	75%
SOLUBILITY IN TRICHLOROETHYLENE	97.72%	---
PVN	+0.26	+0.40
PVN GROUP	AA	AA
P.I. (4°C et 25°C)	1.26	2.18
FLASH POINT (C.O.C.)		---
STORAGE STABILITY	±1°C	---
CHANGE IN MASS	---	0.27%
RETAINED PENETRATION	---	68%

Note: TFOT represents: Thin Film Oven Test

EXAMPLE 4

40 tons of an asphalt composition of the present invention were prepared in the same conditions provided in Example 3, except that the size of the particles of crumb rubber were raised to 4 mesh (about ¼ inch). The test results of this composition are illustrated in Table 4.

TABLE 4

PROPERTIES (TYPICAL)	BEFORE TFOT	AFTER TFOT
PENETRATION 25°C / 100g / 5sec 4°C / 100g / 5sec 4°C / 200g / 60sec	 96.6 dmm 21.0 dmm 57.0 dmm	 61.0 14.5 45.2
VISCOSITY (135°C)	494 mm^2/s	882
SOFTENING POINT	50.5°C	56°C
DUCTILITY (4°C)	8.5cm	5.7cm
PEN 4°C/PEN 25°C	58%	74%
SOLUBILITY IN TRICHLOROETHYLENE	97.72%	....
PVN	+0.1	+0.36
PVN GROUP	AA	AA
P.I. (4°C et 25°C)	1.50	2.07
FLASH POINT (C.O.C.)		...
STORAGE STABILITY	±1°C	...
CHANGE IN MASS	...	0.24%
RETAINED PENETRATION	...	61%

Note: TFOT represents: Thin Film Oven Test

EXAMPLE 5

An asphalt composition was prepared in the same manner as in example 1, except that the selenium was replaced with 0.01 kg of elemental sulfur, and the mixture was agitated for 4 hours at a temperature of 160°C.

EXAMPLE 6

An asphalt composition was prepared in the same manner as in Example 5, except that the elemental sulfur was replaced with a mixture of 0.04 kg of tetramethylthiuram disulfide, 0.04 kg of zinc oxide, and 0.02 kg of stearic acid, and the composition was agitated at 175°C for 4 hours.

EXAMPLE 7

Another composition comprising elemental selenium as the vulcanizing agent was prepared, the amount of selenium being 0.015 kg, and the mixture being agitated for 4 hours at 185°C. The composition was prepared to compare its behaviour with that of the composition of Examples 5 and 6, as illustrated in Table 5 below.

TABLE 5

	Composition of Example 5	Composition of Example 6	Composition of Example 7
Viscosity (mm^2/s)	731	746	750
Softening point (°C)	52	53	52
Ductility (cm)	6.4	5.4	6.3
Penetration (dmm)	83.2	83.2	80.8
Stability high low	52 53	53 54	52.5 53

As it can be seen in Table 5, the vulcanizing agent used had no significant bearing on the properties of the final product. The physical characteristics of the composition of Example 7 are slightly different from those of the composition of Example 1, which is explained by the variations in the time and temperature of agitation after the addition of selenium.

The asphalt composition of the present invention has overcome a serious drawback in the preparation of asphalt composition containing used tires or tubes, in that the composition is homogeneous and very stable, and is produced by an unexpensive process.

While the present invention has been described in connection with specific embodiments thereof, it will be understood that it is capable of further modifications and this application is intended to cover any variations, uses or adaptations of the invention following, in general, the principles of the invention and including such departures from the present disclosure as come within known or customary practice within the art to which the invention pertains, and as may be applied to the essential features hereinbefore set forth, and as follows in the scope of the appended claims.

WHAT IS CLAIMED IS:

1. A process for obtaining a homogeneous asphalt composition comprising the steps of:

a) mixing asphalt with synthetic or natural rubber particles at a temperature of from 160 to 200°C in order to have the particles in suspension in the asphalt;

b) raising the temperature to about 220 to 260°C;

c) injecting air under high pressure;

d) homogenizing the mixture;

e) incorporating a vulcanizing agent and agitating; and

f) recovering the homogeneous asphalt composition.

2. A process according to claim 1, wherein the rubber particles are obtained from waste tires or tubes.

3. A process according to claim 1, wherein the vulcanizing agent is selected from the group consisting of elemental selenium, organo-selenium, selenium salt, elemental sulfur, organo-sulfur and sulfur salt.

4. A process according to claim 1, wherein after step b), the mixture is allowed to stand in at least one reactor for a period of time varying from 1 to 15 hours before air is injected.

5. A process according to claim 4, wherein the air is injected from the bottom of the reactor.

6. A process according to claim 1, wherein the mixture is homogenized by a dispersing device.

7. A process according to claim 1, wherein the rubber particles are obtained from waste tires or tubes and are up to 1/2" thick.

8. A process according to claim 1, wherein an accelerator is incorporated with the vulcanizing agent.

9. A process according to claim 1, wherein the air is injected at a pressure of from about 10 to 50 psi.

10. A process according to claim 1, wherein a petroleum fraction is incorporated in the mixture of step a).

11. A process for obtaining a homogeneous asphalt composition comprising the steps of:

a) mixing from 60 to 99% by weight of asphalt with 1 to 40% by weight of rubber particles obtained from waste tires or tubes at a temperature of from 160 to 200°C in order to have the particles in suspension in the asphalt;

b) heating the mixture at a temperature of about 230 to 250°C;

c) injecting air under high pressure;

d) homogenizing the mixture with a dispersing device;

e) incorporating 0.01 to 3 % by weight of a vulcanizing agent and agitating for about 0.5 to 5 hours; and

f) recovering the homogeneous asphalt composition.

12. An asphalt composition comprising:

- from 60 to 99 % by weight of asphalt;
- from 1 to 40 % by weight of synthetic or natural rubber dissolved in the asphalt; and
- from 0.01 to 3 % by weight of a vulcanizing agent.

13. An asphalt composition according to claim 12, comprising:

- from 80 to 95 % by weight of asphalt;
- from 5 to 20 % by weight of synthetic or natural rubber; and
- from 0.1 to 0.3 % by weight of a vulcanizing agent selected from elemental selenium.

14. An asphalt composition according to claim 12, wherein the rubber is obtained from waste tires or tubes.

15. An asphalt composition according to claim 12, further comprising from 2 to 20 % by weight of petroleum fraction, and/or from 0.01 to 3 % by weight of an accelerator.

16. An asphalt composition according to claim 12, further comprising one or more additives selected from the group consisting of styrene-butadiene-styrene (SBS) and styrene-butadiene-rubber (SBR).

17. An asphalt composition comprising:

- 89.9 % by weight of asphalt;
- 10.0 % by weight of rubber obtained from waste tires or tubes, the rubber being dissolved in the bitumen;
- 0.1 % by weight of elemental selenium.

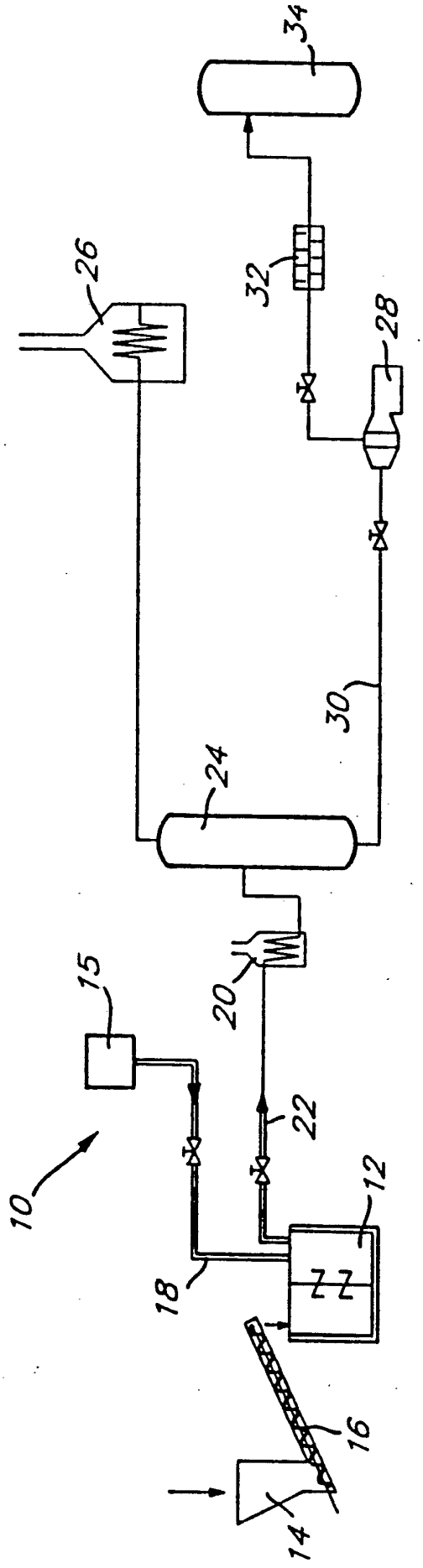

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FIG. 1

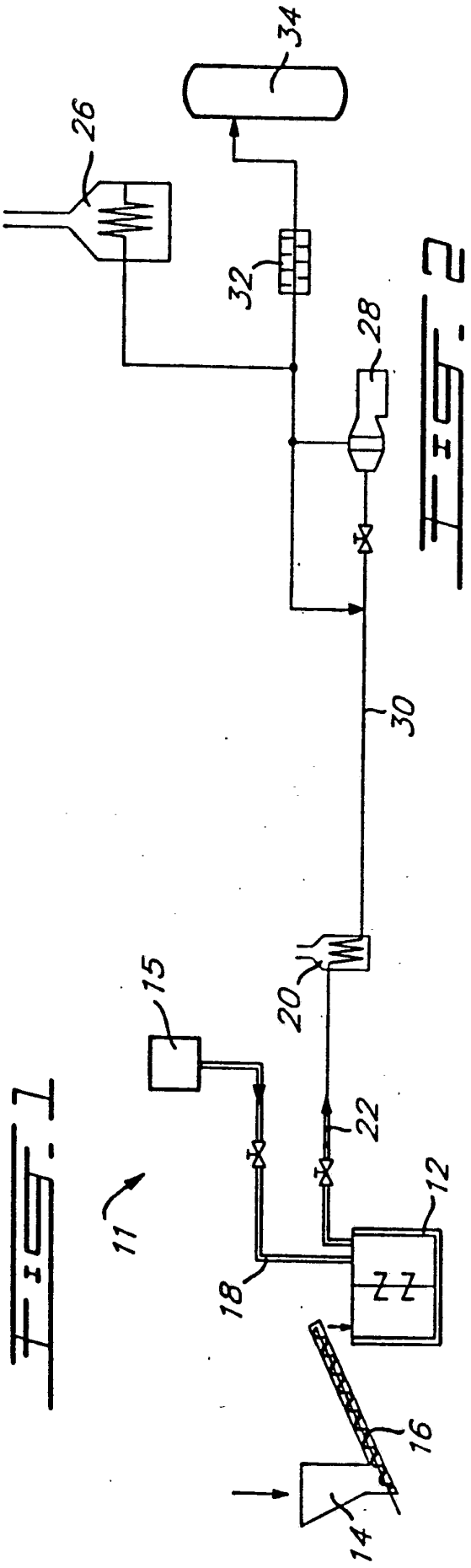

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FIG. 2

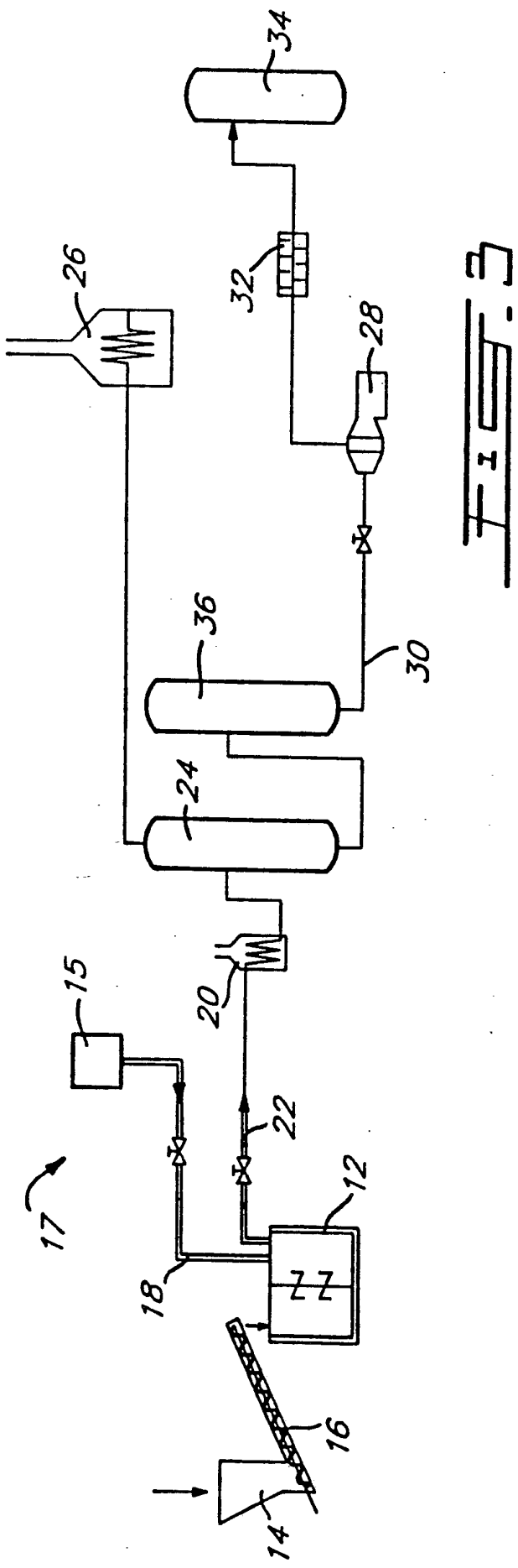


FIG. 3