

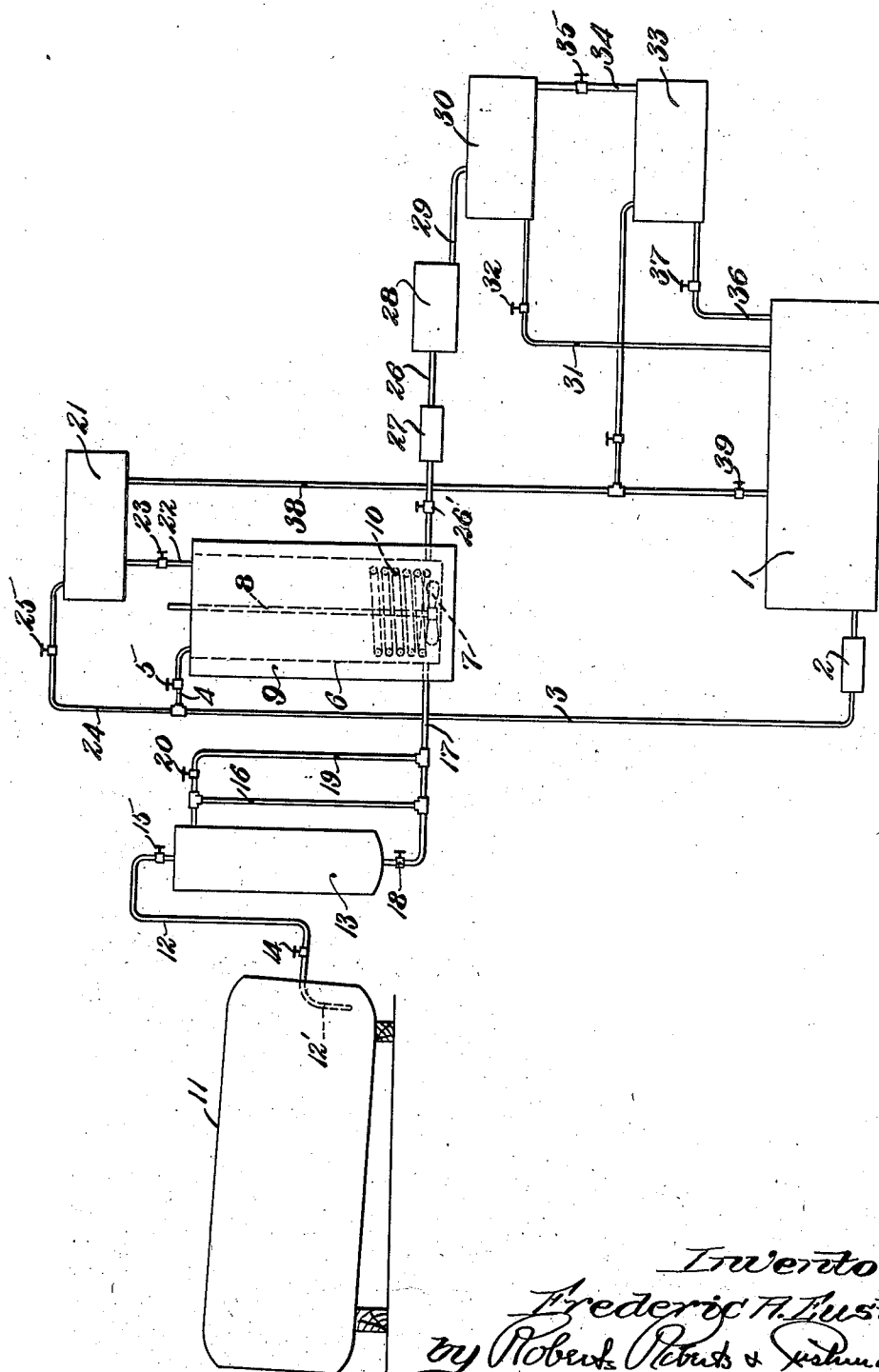
April 28, 1925.

1,535,152

F. A. EUSTIS

APPARATUS FOR REDUCING DYE SOLUTIONS

Original Filed Nov. 17, 1921



Inventor:
Frederic A. Eustis
 by *Robert Roberts & Sherman*
Attys.

UNITED STATES PATENT OFFICE.

FREDERIC A. EUSTIS, OF MILTON, MASSACHUSETTS.

APPARATUS FOR REDUCING DYE SOLUTIONS.

Original application filed November 17, 1921, Serial No. 515,809. Divided and this application filed June 14, 1922. Serial No. 568,164.

To all whom it may concern:

Be it known that I, FREDERIC A. EUSTIS, a citizen of the United States of America, and resident of Milton, in the county of Norfolk and State of Massachusetts, have invented new and useful Improvements in Apparatus for Reducing Dye Solutions, of which the following is a specification.

The present application is a division of an application filed by me Nov. 17, 1921, Serial No. 515,809.

The principal object of the invention is to provide novel and improved apparatus which effects important economies in the process, described in said application, of reducing solutions whose utility in the arts requires that they be used in a reduced state. One of the chief uses to which the invention is applicable is for reducing indigo dyes and other vat dyes, and by way of illustration I will therefore describe the apparatus and process as applied to the reduction of an indigo dye solution, but it is to be understood that the invention is not limited to the reduction of indigo, but is also applicable to the reduction of other solutions.

To dye cloth or yarn with indigo, it has been customary to maintain a solution of reduced indigo in one or more vats through which the cloth or yarn is passed. The solution of indigo is absorbed by the cloth or yarn, and after the material leaves the vat the indigo is oxidized by exposure to the air, and is precipitated as an insoluble coloring matter in the material.

In order to maintain the liquor in suitable condition for use it is necessary to feed to it from time to time proper quantities of indigo paste or dye and of a reducing agent, which reducing agent may be supplied in the form of a solid or liquid. Sufficient reducing agent must be added to thoroughly reduce the indigo which is added, and also some further quantity to make good any oxidation losses that occur during the process of dyeing by reason of oxidation on the surface or from oxygen which is carried into the solution in the cloth or water or otherwise. Sufficient reducing agents, particularly when in solution form, are of considerable volume, and since there is nothing normally taken out of the vat excepting the slight amount of coloring matter which ad-

heres to the cloth, the vats generally overflow or require to have a part of the liquor drawn off as a waste product to prevent overflow. The cloth or yarn is usually introduced wet and is taken out with about the same volume of moisture, so that it neither adds much to nor subtracts much from the volume of liquor in the dye vat.

The reducing may be done with dry sodium hydrosulphite or with a solution of this salt, or other less efficient reducing agents. The dry hydrosulphite has the advantage that it is easy to handle, efficient, and of small volume, but it is expensive and by its nature probably always will be. A solution of hydrosulphite, while procurable at much less cost than the solid salt, has the disadvantage already noted of adding bulk to the liquor to be reduced, thereby causing overflow and waste. Other reducing agents are bulky and are generally less efficient, and give generally less good results.

With the present invention a reducing agent is produced in solution form and is made in a part of the dye solution or other solution to be reduced, thereby securing the advantage of producing a hydrosulphite in solution form, but eliminating the trouble, waste and loss due to overflow or to an increase in volume of the liquor heretofore mentioned, and otherwise securing economies in the reduction of the dye solution.

The accompanying drawings illustrate diagrammatically apparatus embodying the invention, and adapted for carrying out the said process which is claimed in my aforesaid application.

Referring to the drawings, 1. represents a vat or vessel containing the dye liquor or other solution to be treated. The solution is pumped from the vat 1 by pump 2 through pipe 3 and pipe 4 controlled by valve 5 into the mixing tank 6 where the hydrosulphite solution is formed. In the process being described by way of illustration, this dyeing liquor consists of a reduced indigo solution more or less oxidized by the dyeing process and other conditions of its use. An agitator 7, consisting of a paddle or propeller arranged near the bottom of the mixing tank and driven by a shaft 8, is then started and there is added to the liquor in the mixing tank a suitable quantity of zinc dust. Other powdered metals such as iron might

be used but powdered zinc is preferable. The reactions which take place in the mixing tank generate heat and in order to avoid overheating the solution, the mixing tank 6 is cooled by a water jacket 9 or by cooling coils 10, or both. There is then introduced into the mixing tank a suitable quantity of liquid sulphur dioxide from the storage tank 11. A pipe 12 joined to an extension 12' leading to the lowermost point within the sulphur dioxide container 11 connects with the top of the measuring tank 13, which is the supply tank for delivering a measured quantity of sulphur dioxide to the mixing tank. Suitable valves, as 14 and 15, may be provided for conveniently controlling the pipe 12. The amount of sulphur dioxide used with each batch of material in the mixing tank is measured either by weight or by volume in the measuring tank 13. If the sulphur dioxide is to be measured by weight, the tank 13 will be mounted on scales; if it is to be measured by volume the tank 13 may be provided with a gauge glass 16 with which is associated a suitable measuring scale to indicate the quantities contained in the tank at various levels.

A uniform flow of sulphur dioxide into the mixing tank 6 is secured by passing it from the bottom of the measuring tank 13 through a short pipe or hose 17 which is made of such proportions that the loss of head caused by the flow of the liquid through the pipe 17 at the desired rate will balance the pressure in the measuring tank, which pressure is a function of the temperature of the room. The connection 17 is controlled by a valve 18 at the bottom of tank 13. In case the pressure in tank 13 becomes excessive, it may be reduced by opening the valve 20 in a by-pass pipe 19, and closing the valve 18, which allows the escape of gas through pipes 19 and 17, thus directly relieving the pressure in tank 13 and also reducing the temperature and consequently the pressure in tank 13 by the volatilization resulting from opening the by-pass 19. The sulphur dioxide is introduced through pipe 17 near the point in the mixing tank where the agitation of the solution is most active. The zinc or other metallic dust is kept in suspension in the solution by the action of the agitator and the sulphur dioxide as it enters meets a swirling mass of zinc dust and liquid so that no excess of sulphur dioxide will occur at any point.

The sulphur dioxide reacts with the zinc dust in the dye liquor and forms first zinc hydrosulphite. This probably reacts at once with any unreduced indigo in the solution.

After the sulphur dioxide is all introduced a suitable quantity of alkali such as caustic soda or carbonate of sodium is added to the mixing tank to convert the zinc hydrosulphite and other zinc salts present into

sodium salts, and also to hold the indigo in solution. The dye liquor often contains an excess of caustic alkali which should be taken into account in determining the amount of caustic alkali to be added. The caustic soda or carbonate of sodium or other alkali may, if desired, be put directly into the mixing tank in dry form and there taken into solution. The solution of the caustic alkali liberates a considerable quantity of heat, but the cooling coils and water jacket will be made ample to absorb this.

Instead of introducing the alkaline agent dry into the mixing tank it may, if preferred, be introduced in solution. To this end a suitable tank 21 is provided in which the alkaline agent is dissolved in part of the dyeing solution supplied from vat 1 through pipe 3 and pipe 24 controlled by valve 25. The alkaline solution is delivered in the desired quantity to the mixing tank 6 from tank 21 through pipe 22 controlled by valve 23.

As the alkali is added the zinc is precipitated. The solution in the mixing tank 6 is then forced through pipe 26 controlled by valve 26' by means of a pump 27 into a filter press 28 which may be of usual or suitable form, and from the filter press 28 the clear solution carrying an excess of the reducing agent with the indigo all thoroughly reduced, is run by pipe 29 into a storage tank 30.

Since the precipitate retained in the filter press will contain a small amount of available reducing solution, a further part of the dye liquor may be pumped from the dye vat 1 through the mixing tank 6 and through the filter press 28 to wash these precipitates. This liquor will absorb substantially the last traces of the reducing agent. It may be followed by a small quantity of water merely sufficient to replace the solution retained by the precipitate. The press may then be cleaned and set up ready for the next run.

The reducing solution collected in the storage tank 30 may then be used in two ways. Part of it may be run directly into the dye vat 1 through pipe 31 controlled by valve 32. Part of it may be run into the indigo mixing tank 33 through pipe 34 controlled by valve 35. In the indigo mixing tank 33 suitable quantities of fresh indigo paste or dye are added together with suitable quantities of caustic soda. These ingredients when thoroughly mixed will then be passed through pipe 36 controlled by valve 37 into the dye vat 1.

As an alternative procedure, the storage tank 30 and the indigo mixing tank 33 might be omitted and the solution delivered direct from the mixing tank 6 through the filter press 28 into the dye vat 1. In this event the required additional quantities of indigo paste and of caustic may be added in the

mixing tank 6 after the hydrosulphite solution is formed with sulphur dioxide and zinc dust, or the indigo paste and the caustic might be added directly in the dye vat 1, caustic being supplied, if desired, from the tank 21 through pipe 38 controlled by valve 39.

With the foregoing apparatus and process a part of the solution itself which is to be reduced is brought in contact with the hydrosulphite while the latter is being produced; that is, the hydrosulphite is manufactured with sulphur dioxide and zinc dust or its equivalent directly in a part of the solution to be reduced by it, thus avoiding oxidation and waste, and making it possible to use liberal quantities of solution from the dye vats and thereby to work with a solution of convenient dilution without the disadvantage of increasing the volume of the dye liquor and thereby necessitating either the waste of a part of the liquor or a treatment of it by an expensive reclaiming process. Moreover, the apparatus and process described minimizes the mechanical handling of the solutions and salts and makes possible a procedure on a large scale with a minimum cost for labor.

The combination of the use of the dye liquor and liquid sulphur dioxide in conjunction with zinc or other suitable metal for the production of hydrosulphite solutions of reducing agents, has an especial advantage because the liquid sulphur dioxide is easily available in a very pure condition and carries into the dye liquor a minimum of impurity, whereas the introduction of sodium bisulphite for example into the dye liquor for the production of a reducing solution is undesirable because with sodium bisulphite there is introduced so much soluble salts as to be detrimental to the dyeing process and to render the same impracticable.

I claim:

1. Apparatus for reducing solutions comprising a vat, a mixing tank, means to deliver solution from the vat to the mixing tank, a sulphur dioxide supply tank, means to deliver sulphur dioxide from the supply tank to the mixing tank, a filter, means to deliver solution from the mixing tank to the

filter and means to deliver filtered solution from the filter to the vat.

2. Apparatus for reducing dye solutions comprising a dye vat, a mixing tank, means to deliver solution from the dye vat to the mixing tank, a filter, means to deliver solution from the mixing tank to the filter, a storage tank, means to deliver filtered solution from the filter to the storage tank, a dye mixing tank, means to deliver solution from the storage tank to the dye mixing tank, means to deliver solution from the storage tank to the dye vat, and means to deliver solution from the dye mixing tank to the dye vat.

3. Apparatus for reducing solutions comprising a vat, a mixing tank, means to deliver solution from the vat to the mixing tank, a sulphur dioxide supply tank, means to deliver sulphur dioxide from the supply tank to the mixing tank, an alkali tank, means to deliver solution from the vat to the alkali tank, means to deliver alkali solution from the alkali tank to the mixing tank, a filter, means to deliver solution from the mixing tank to the filter, and means to deliver filtered solution from the filter to the vat.

4. Apparatus for reducing dye solutions comprising a dye vat, a mixing tank, means to deliver solution from the dye vat to the mixing tank, a sulphur dioxide supply tank, means to deliver sulphur dioxide from the supply tank to the mixing tank, an alkali tank, means to deliver solution from the dye vat to the alkali tank, means to deliver alkali solution from the alkali tank to the mixing tank, a filter, means to deliver solution from the mixing tank to the filter, a storage tank, means to deliver filtered solution from the filter to the storage tank, a dye mixing tank, means to deliver solution from the storage tank to the dye mixing tank, means to deliver solution from the storage tank to the dye vat, and means to deliver solution from the dye mixing tank to the dye vat.

Signed by me at Boston, Massachusetts this 7th day of June 1922.

FREDERIC A. EUSTIS.