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MATERIAL FOR SELECTIVELY CONTROLLING METAL PICKLING IN ACID BATHS

No Drawing.

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This invention relates generally to metal pickling and especially to that pickling where steel is cleaned in a bath of sulphuric or other acid.

- 5 The objects of the invention are to provide an improved selective controlling material for metal pickling, to save acid, to save metal, to prolong the life of a pickling bath, to reduce acid brittleness, to minimize
10 blisters, to prevent over-pickling, to reduce the roughness of the pickled work, to improve the appearance of the pickled work and generally to improve the art of pickling metal. It is a further object of the invention
15 to provide a material for carrying out the above objects that is easily transported and otherwise handled, that is economical to use, and which, when used according to the following specifications, will not injure health
20 nor effect the skin.

- To free steel of scale and rust it is immersed in a bath of dilute acid, usually sulphuric acid, although hydrochloric acid and even phosphoric acid are sometimes used. The
25 acid dissolves or otherwise removes these coatings but will also attack the clean metal and thus unnecessarily causes much loss of good metal and good acid. In order to overcome this waste, controlling agents are added
30 to the acid bath rendering it selective in its action so that during the cleaning operation, usually called pickling, the action of the acid is confined to cleaning thus preventing to a
35 great extent the attack of the acid on the clean metal.

- Heretofore it has been my opinion that in order for a substance to be effective as a selective controlling agent it must of necessity be
40 soluble in the pickling bath and, hence, in my search for better and cheaper controlling agents I was restricted to materials of that class. I have discovered, however, that it is not necessary that the material be soluble,
45 provided it can be divided into particles of sufficient fineness to remain suspended, in the pickling bath. I have discovered that this can be accomplished by triturating the selective control material with a buoying
50 agent and then adding the admixture to the pickling bath, the buoying material acting

to keep the fine particles of the controlling agent suspended and permit it to be diffused throughout the bath by the natural circulation occurring in the pickling operation.

I have found that substances which apparently had no selective controlling properties, when added directly to the pickling bath, show marked effects when used according to my invention; as for instance, anhydroformaldehydeaniline, "thionex", diorthotolylthiourea, diparatolylthiourea, dimetatolylthiourea and thiocarbanilide. It will be noted that these materials are in that class known as "rubber accelerators" which are organic compounds containing sulphur and nitrogen. They are solid bodies which are substantially insoluble in water. When, however, they are triturated and thereby very finely divided and are mixed with a buoying agent they will then when added to the bath act very effectively to modify the action of the acid bath in the desired manner. I find the best effects are obtained by triturating the controlling agent in the buoying agent.

I have found that the best buoying agents are colloidal materials, such as evaporated cellulose pulp waste liquor, starch, flour, clay, etc. Of these, I prefer to use the evaporated cellulose pulp waste liquor, due to its economy and effectiveness.

To carry out my invention commercially, I may admix in the following proportions:

	Pounds
Diorthotolylthiourea	2.00
Evaporated cellulose pulp waste liquor	3.00

These proportions may be varied to a great extent keeping in mind however that sufficient of the colloidal material should be used to buoy up the quantity of the selective controlling material employed.

This admixture is then thoroughly ground, agitated or beaten so as to forcibly bring the two ingredients into intimate contact. In other words, I triturate the admixture. It is then ready to be introduced into the pickling bath. The quantity required for selectively controlling the pickling of steel will vary according to the degree of controlling effect desired, but a pickling bath of the following

proportions will be found satisfactory for pickling steel if operated at 180° F.:

Water -----	500.00 gallons.
Sulphuric acid (66°) -----	25.00 gallons.
5 Triturated control material--	1.00 pound.

As the bath is used, more and more of the acid becomes exhausted and it is necessary to reinforce the bath with fresh acid from time to time. I have found that it is well to proportionally add additional selective controlling material as fresh acid is added, thus maintaining a constant relation between the amount of acid and the amount of selective control.

As one of the ingredients of my selective controlling material is colloidal in nature, it is often found that a foam will be produced on the pickling bath. This foam has the advantage of holding the hydrogen, which results from the action of the acid on the metal, long enough for it to deposit its moisture before it escapes to the air. This eliminates the spraying of acid into the surrounding air, to the great benefit of health and property. The foam, however, has a disadvantage, in that it is apt to trap additions of selective controlling materials and so prevent them reaching the bath, unless care is exercised to brush aside the foam, when adding the material. I have found that this difficulty may be overcome by compressing my controlling material into the form of briquettes, which have sufficient weight to penetrate the foam and enter the bath, and there dissolve. In order to give the briquette a desired size, filling material may be added to my triturated admixture. Such materials as sodium chloride, sodium sulphate, sugar, etc., may be employed. I prefer to employ sodium chloride when using a sulphuric acid pickling bath, as this generates hydrochloric acid in the bath and tends to improve the appearance of the resulting work. The following proportions will be found satisfactory for a material that may be briquetted:

	Pounds
Diorthotolylthiourea -----	2.00
Evaporated cellulose pulp waste liquor--	3.00
50 Sodium chloride-----	5.00

more or less as experience may dictate.

The first two ingredients are first mixed and triturated and then the filler added and thoroughly mixed to distribute it uniformly. The resulting admixture may be used as is, in the powdered form, or it may be briquetted. The briquetting may be accomplished with the dry material but better results are obtained when the admixture is slightly moistened with water.

The advantages of my invention can be readily demonstrated when pickling steel in a bath of sulphuric acid. Under like conditions an uncontrolled bath will operate for perhaps thirty minutes before it becomes ex-

hausted and has to be reinforced with additional acid. If the selective controlling material and the buoying agent are simply mixed before adding them to the bath or if the two are added separately to the bath, the life of the bath is not materially prolonged, it may operate for thirty-five minutes. However if the ingredients are first mixed and then triturated, the bath will have a life of about eighty minutes. In other words, more than twice the amount of work may be pickled with the same acid when my invention is employed. As the amount of my selective controlling agent used is relatively small and inexpensive, the net savings effected are evident. In addition to this economy, the quality of work produced is improved by my invention in that the danger of over-pickling, in an endeavor to remove obstinate patches of scale, is avoided and the amount of hydrogen resulting from the pickling operation is greatly reduced, which acts to reduce blisters and lessen acid brittleness.

The briquetting of my selective controlling material not only enables it to penetrate the foam on the top of the pickling bath, but it serves to further unite the control material and colloidal material and also affords a simple and effective way of measuring and proportioning the selective controlling material when adding it to the bath, thus securing uniform results.

Having now described my invention, I claim:—

1. A material for selectively controlling the action of a metal pickling bath comprising a triturated admixture of a water insoluble controlling agent and a colloid, said admixture being adapted to be suspended in the liquid thereof and circulated through the bath by the natural circulation thereof.

2. A material for selectively controlling metal pickling baths comprising a triturated admixture of a water insoluble selective controlling material and a buoying agent therefor consisting of cellulose pulp waste liquor.

3. A material for selectively controlling metal pickling baths comprising a triturated admixture of a water insoluble selective controlling material and a buoying agent for the said selective controlling material.

4. A material for selectively controlling metal pickling baths comprising a triturated admixture of diorthotolylthiourea and a buoying agent for the said diorthotolylthiourea.

5. A material for selectively controlling metal pickling baths comprising a triturated admixture of diorthotolylthiourea and evaporated cellulose pulp waste liquor.

6. The method of preparing a metal pickling bath comprising triturating together a water insoluble selective control agent and a colloid, and thereafter introducing said admixture into the bath.

7. The method of preparing a selective control material for use in metal pickling baths comprising triturating together a water insoluble selective control material and a buoying agent therefor consisting of cellulose pulp waste liquor.

8. The method of preparing a selective control material for use in metal pickling baths comprising triturating together a water insoluble selective control material and a buoying agent therefor.

9. The method of preparing a selective control material for use in metal pickling baths comprising triturating together diorthotolylthiourea and a buoying agent for the said diorthotolylthiourea.

10. The method of preparing a selective control material for use in metal pickling baths comprising triturating together diorthotolylthiourea and evaporated cellulose pulp waste liquor.

Signed at New York, in the county of New York and State of New York, this 1st day of October, A. D. 1928.

JAMES H. GRAVELL.