

A SIMPLE METHOD OF RECOVERY OF MINERAL OIL FROM WASTE CUTTING OIL EMULSIONS

by

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ABSTRACT

A simple chemical method for recovery of mineral oil from waste cutting oil emulsions using a chemical which is wholly indigenous has been standardised. It has been established that requisite volume of 15% alum solution at 50°C results in complete oil separation irrespective of the nature of oil originally used for making the emulsions and the resulting emulsion composition. It has been found that the separated mineral oil is suitable for use as furnace fuel oil. A lay out of the plant required has been suggested and a pre-construction cost estimate has been presented.

Introduction

Metal working is one of the most important industrial operations. Depending on the accuracy of manufacture, the speed of production and the materials being handled, different metal working oils are used as lubricants. One of the oils used is known as "soluble oil" which is used in the form of an emulsion with water. After use over a period depending on the severity of operating conditions, the stability of the emulsions is impaired and the emulsions accumulate fine particles and thus become unfit for further use. The used emulsions are then disposed of as such.

The Defence Services in India alone are importing soluble oil of the above type to the tune of about 6500 gallons annually. Although the used emulsions are unfit for use, nevertheless the practice of disposing of the emulsions as such, prepared from material imported from abroad, is not considered economical as there is a scope for separating the oil component from such emulsions in good yield by simple chemical means and utilisation of the separated oil for some alternative use. It is, therefore, desirable to develop a simple method for recovery of oil from waste emulsions and to find out an alternative use of the separated oil. It is felt that quite apart from the problem of sewage disposal, this will ensure better utilisation of a product originally procured from foreign sources. The present work describes a simple chemical method for recovery of oil from waste emulsions.

Details of Plants

A line diagram of the plant needed, together with complete details of plants are given in Appendix I.

Method for Coagulation and Settling

The following method has been finally standardised for coagulation/settling after large number of experiments. The numerals refer to the diagram in Appendix I.

- (i) The waste emulsion is allowed to settle undisturbed in the settling tank (1) overnight to allow the free oil to separate at the top and the fine sludge to settle at the bottom.
- (ii) The clear emulsion is transferred to the flocculation tank (5) and alum solution of requisite concentration is added to the emulsion at the appropriate temperature under stirring (stirring speed 300 r.p.m.) and the stirring is continued for 5 minutes after the addition of alum solution is complete. The stirring is then discontinued.
- (iii) The separated water is then drawn off and thrown away and the hazy oil collected and centrifuged (11) for 15 minutes.
- (iv) The clear oil from the centrifuge (11) is transferred to a container.

Preconstruction cost estimate

This has been given in Appendix II.

Results*(i) Oils used.*

Three different brands of oils have been used. The characteristics of the oils used are given in Table I.

(ii) The Characteristics of the alum used.

This is given in Table II.

(iii) The effect of alum dilution vs emulsion composition on efficiency of oil separation.

The results are given in Table III.

(iv) The relationship between oil composition, emulsion composition and efficiency of oil separation.

The results are given in Table IV.

(v) The effect of temperature on oil separation.

The results are given in Table V.

(vi) The effect of alum concentration on oil separation.

The results are given in Table VI.

(vii) The characteristics of the oils separated from the waste emulsions.

The test-data are given in Table VII.

TABLE I
Characteristics of the Oils used

Tests	Oil A	Oil B	Oil C 3
(i) Viscosity (kinematic) at 100°F, CS	43.17	52.84	41.74
(ii) Ash, %	1.5	1.2	1.3
(iii) Oil content of 10% emulsion in water (IP)* ..	9.6	9.7	9.7

TABLE II
Characteristics of the Alum used

(i) Potash alum ($Al_2(SO_4)_3, K_2SO_4, 24(H_2O)$), % by weight ..	99.0
(ii) Soluble iron compounds (as Fe), % by weight	0.005
(iii) Water insolubles, % by weight	0.20
(iv) Free Acid (as H_2SO_4), % by weight	0.09
(v) Ammonia (NH_3), % by weight	0.20

TABLE III

The effect of alum dilution *vs* emulsion composition on efficiency of oil separation.

Oil used is 'A'. Strength of the alum solution used is 10% (w/v). Test Temperature is 25°C (room temperature).

Emulsion composition ➤	10 ml. oil : 300 ml. water			10 ml. oil : 200 ml. water			10 ml. of oil : 100 ml. water		
	22	11	5.5	22	11	5.5	22	11	5.5
Alum solution used, ml. ➤									
Period observation taken ▼	ml. oil separated ▼								
1 minute	3	2	7	2	3	8	4	6	8
2 minutes	5	5	..	5	5	..	7	8	9
3 minutes	5	..	7	7	..	9	8	10
4 minutes	5	..	7	7	..	9	8	..
5 minutes	10	5	10	7	8	10	10	9	..

NOTE—The separated oil is hazy in appearance.

TABLE IV

The relationship between oil composition, emulsion composition and efficiency of oil separation.

Oils used are A, B and C. Strength of alum solution used is 10% (w/v). Vol. of alum solution used is 5.5 ml. Test Temperature is 25°C (room temperature).

Emulsion composition →	10 ml. oil : 300 ml. water			10 ml. oil : 200 ml. water			10 ml. oil : 100 ml. water		
	ml. oil separated from ∇								
Period observation taken ∇	Oil A	Oil B	Oil C	Oil A	Oil B	Oil C	Oil A	Oil B	Oil C
1 minute	5	7.5	4	8	6	6	9	..	8
3 minutes	5	10	10	..	10	10	10
5 minutes	10	10	9	10

NOTE—The separated oil is hazy in appearance.

TABLE V

Effect of temperature on oil separation.

Oils used are A, B and C. Strength of Alum Solution used is 10% (w/v). Composition of emulsion is 10 ml. oil : 100 ml. water. Test temperature is 50° ± 1°C.

Period after which observation taken ∇	ml. oil separated—using 5.5 ml. of alum soln.		
	Oil A	Oil B	Oil C
1 minute	9	4	9
2 minutes	10	6	10
3 minutes	8	..
5 minutes	10	..

NOTE—The separated oil is hazy in appearance.

TABLE VI

Effect of alum concentration on oil separation.

Oils used are A, B and C. Composition of emulsion used is 10 ml. oil : 100 ml. water. Test temperature is $50^{\circ} \pm 1^{\circ}\text{C}$.

ml. of alum solution used➤			5.5			5.5		
			10% (w/v)			15% (w/v)		
Strength of alum solution➤			ml. oil separated from			ml. oil separated from		
			Oil A	Oil B	Oil C	Oil A	Oil B	Oil C
Period observation taken▼			Oil A	Oil B	Oil C	Oil A	Oil B	Oil C
1 minute	9	4	9	7	8	7
2 minutes	10	6	10	10	10	10
3 minutes	8
4 minutes
5 minutes	9

NOTE—The separated oil is hazy in appearance.

TABLE VII

Characteristics of separated/centrifuged oil from waste emulsion.

Oils used for making emulsion are A, B and C. Particulars to which tested are Ind/SL/QMG/4309(a) for furnace fuel oil.

Test Report

Tests	Oil separated from emulsion prepared from			Specification requirements
	Oil A	Oil B	Oil C	
(i) Appearance	clear and bright			Shall be clear and bright.
(ii) Viscosity (Kinematic) at 100°F	32.68 cs	36.38 cs	38.66 cs	Not less than 49.2 cs
(iii) Inorganic acidity	Nil			Nil
(iv) Flash Point (P & M), closed, °F	above 500°	290°	335°	Not below 149°F
(v) Ash, %	0.58	0.54	0.51	0.05 maximum
(vi) Sulphur corrosive (100°C for 3 hrs; I.P.)	Passes			Copper strip shall show no greater discoloration than that shown by a similar strip exposed to dry heat at the same temp. and period.
(vii) Calorific value (gross; I.P.)*	22,300	22,570	18,000	18,500 B. Th. U. minimum.
(viii) Total sulphur (I.P.)*, % ..	0.79	0.82	1.1	2.5% maximum
(ix) Water (I.P.)*, %	1.6	1.8	1.5	1.0% maximum

NOTE—*I.P. refers to the latest standard methods of tests published by the Institute of Petroleum, U.K.

Discussion

Aluminium sulphate (alum) is extensively used for sewage disposal¹. The process has, therefore, been standardised using alum solution. The quality of alum utilised is such that it can be procured indigenously. In order to collect a comprehensive data, three different brands of oil have been used in this investigation. A 10% alum solution has been used initially purely on 'ad-hoc' basis to standardise the process.

At first, the effect of different volumes of alum solution of the above strength vis-a-vis emulsion composition on the efficiency of oil separation has been investigated. The results are given in Table III & IV. It is evident that irrespective of the nature of the oil used for preparing the emulsions and the composition of the prepared emulsion, optimum oil separation can be effected at a very short time using only 5.5 ml. of the alum solution of above strength. It is also clear that richer the emulsions in respect of oil content, the better is the tendency for the oil to separate. In view of this finding, all subsequent experiments have been carried out using 5.5 ml. of alum solution.

The effect of higher temperature using fixed volume of alum solution as stated above on oil separation is given in Table V. Data regarding oil separation efficiency using higher concentrations of alum solution at elevated temperatures have been presented in Table VI. From these and from the characteristics of the oil given in Table 1, it follows that practically complete oil separation from an emulsion containing 10 cc of oil results in a very short time at 50°C when 5.5 ml. of 15% alum solution is used. It would, therefore, be necessary to determine initially by laboratory test the oil content of the used emulsions with a view to ascertaining the volume of alum solution required for optimum oil separation. However, the separated oil in all cases is hazy and has to be centrifuged to obtain clarity of the desired degree.

From the data given in Table VII, it can be concluded that the separated oil can be utilised as furnace fuel oil. It should be pointed out that the separated oil may not completely meet the requirements of the relevant Defence Specification. The use being not of critical nature, these deviations can be ignored.

The details of plant required have been presented in the Appendix I. It would be observed that the plant needed is easy to procure/fabricate and not costly. The method of separating the oil is simple, does not require any rigid technical control and the process can be carried out casually on as-required-basis. From the preconstruction cost estimate presented in Appendix II, it would appear that the oil can be separated at a negligible cost provided the waste emulsion is fairly rich in oil content. In fact, the cost of the separated oil is cheaper than the freshly purchased furnace oil. The process seems to be uneconomical in the case of dilute emulsions. Considering Defence consumptions only, the resulting saving in foreign exchange does not seem to be significant, but it is presumed that on a national basis the saving can be substantial.

It should be pointed out that utilisation of the separated oil as furnace fuel oil is not very desirable. A better use would be to incorporate a suitable

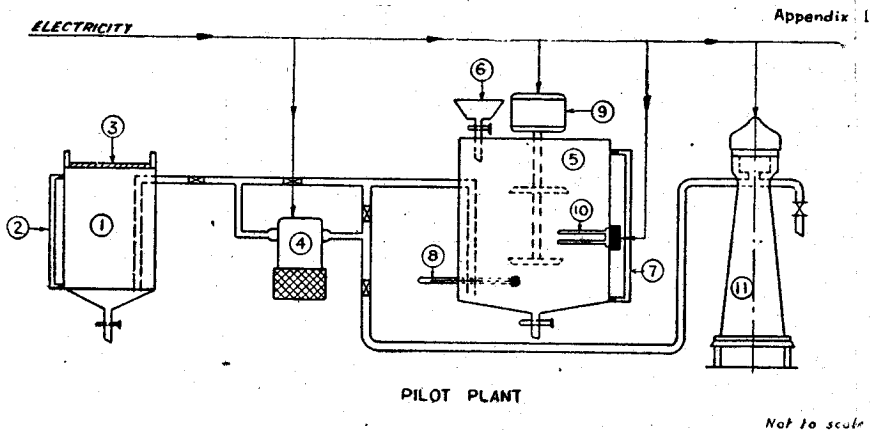
indigenous emulsifier in the separated oil and to use the blended oil for the purpose for which it was originally intended. Such a work is worth undertaking.

Acknowledgement

Our thanks are due to Dr. Kartar Singh, Director, for his kind interest.

Reference

- 1 Babbit H. E., *Sewrage and Sewage Treatment* (Wiley, Ny. 1947 6th Edition).



(Waste emulsion before and after addition of alum solution)

APPENDIX I—contd.

Item No. in Appendix	1, 2 & 3	4	5, 6, 7, 8, 9 & 10	11
Equipment	Settling Tank	Centrifugal pump	Treatment Tank	Centrifuge
Function	Settling of dirty emulsion	Pumping the emulsion	Coagulation/settling the emulsion/oil	Centrifuging the hazy oil
Description	<p>1. Rectangular, capacity—6 cu. ft. material of construction—mild steel.</p> <p>2. Side gauge, glass.</p> <p>3. Sieve, mesh size 60, material of construction—mild steel.</p>	<p>Standard make Cap. 1 H.P.</p>	<p>5. Rectangular capacity 8 cu ft. material of construction—mild steel.</p> <p>6. Inlet for alum solution</p> <p>7. Side gauge, glass</p> <p>8. Thermometer</p> <p>9. Stirrer and Stirrer motor, capacity of the motor—0.5 H.P. standard make.</p> <p>10. Immersion heater, capacity—1 KWH</p>	<p>Bottle type, vertical perforated basket, batch-centrifuge with an unloader for bottom discharge. The basket is 3" deep contains 2 square feet of filtering area and a lip of 2". Speed 1400 R.P.M.</p>
Electricity	..	0.75 KWH	1.5 KWH	1 KWH

NOTE—1" dia standard pipe to be used for connection.

APPENDIX II

PRECONSTRUCTION COST ESTIMATE FOR A SMALL PLANT (CAPACITY 50 GALLONS) FOR RECOVERY OF MINERAL OIL FROM WASTE CUTTING OIL EMULSION

1. Raw material's cost (Basis 1 hr/day operating)

	Rs. nP.
Alum, Potash (2 kgm/50 gall. of emulsion)	1.40

2. Service cost (Basis 1 hr/day operating)

Electricity 1.25 KWH at 25 nP per KWH	0.31
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Total daily cost	1.71
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Total monthly cost (25 days/month)	42.75
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3. Equipment cost summary

(a) Centrifuge (No. 1)	2500.00
(b) Settling Tank (No. 1)	150.00
(c) Treatment Tank (No. 1) with motor driven stirrer and immersion heater	500.00
(d) Centrifugal Pump	800.00
(e) Installation, instrument, miscellaneous fittings etc. (5% of equipment charge)	200.00

Total	4150.00
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4. Labour cost (1 hr/day operating)

(a) Labour (one) at Rs. 100 per worker/month (8 hrs working/day for 25 days/month)	0.50
(b) Supervision (20% of labour)	0.10
(c) Overhead (100% of labour)	0.50

Daily cost	1.10
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Total monthly cost (25 days/month)	27.50
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5. Capital Investment

(a) Equipment	4150.00
(b) Land & Building*	NIL
(c) Fixed Charges (1.0% of (a) & (b))	41.50

Total	4191.50
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* NOTE—As the plant is not going to occupy considerable space it has been assumed that the plant can be accommodated in the existing factory site undertaking reclamation and no new land/building is required).

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6. Monthly recurring expenses inclusive of fixed charges but exclusive of capital investment

	Rs. nP.
(a) Raw Material & Service	42.75
(b) Labour and overhead	27.50
(c) Fixed charge	41.50
Total monthly cost ..	111.75

7. Estimated quantity of oil separated/month

(i) Basis:—emulsion composition 15% oil & 85% water	187.5 gallons
(ii) Basis:—emulsion composition 3% oil & 97% water	37.5 gallons

**8. Estimated cost per gallon of separated oil
(exclusive of Capital investment)**

	Rs. nP.
(a) Basis:—emulsion composition 15% oil & 85% water	0.59
(b) Basis:—emulsion composition 3% oil & 97% water	2.93

9. Cost of freshly purchased furnace fuel oil

Furnace fuel oil per gallon	0.76
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