# *Regeneration of Spent Limonite using Gasoline. I*

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It was observed that the crude gasoline, a by-product in coal gasification process, could dissolve certain quantity of sulfur when hot, from the spent limonite and the dissolved sulfur could easily be crystallized out by cooling. This observation suggested that it could be utilized profitably as a solvent for sulfur. Some important characteristics of this gasoline were studied including the solubility of sulfur in it. On satisfactory preliminary tests, a batch process of regeneration of limonite was tried. It was possible to recover 90.9% sulfur from spent limonite at a regeneration temperature of 85°C using a weight ratio 11.7 : 1 of gasoline and spent limonite respectively.

Next, a continuous flow of hot gasoline through a fixed bed of spent limonite was tried. This had shown some improvement over the batch process mentioned above, 96.0% sulfur was recovered at 86°C with gasoline limonite ratio 12 : 1 (w/w). The main difficulty encountered in this process was the sticking of sulfur crystals on the cooling surface and thereby heat transfer was retarded. This was overcome by direct quenching in cold water. Another problem was to remove the retained gasoline from the regenerated limonite. The removal was effected by passing nitrogen through the limonite bed in suit at regeneration temperature. The exit gas was then washed with oil to recover gasoline. The over-all activity of this regenerated limonite was found to be over 80.0%.

It has already been discussed in part I¹⁾ and part II²⁾ that the recurring import of fresh limonite and the disposal of spent limonite are problems to this factory. A practical method²⁾ (cf. part II) for the production of limonite from iron ore has since been developed. Relatively easier method of solvent extraction was also considered but due to non-availability of indigenous solvent it was not tried earlier. In the meantime, the problem of by-product gasoline purification was at hand. The crude gasoline could not be marketed for its high sulfur content in the form of $H_2S$, mercaptans and other organic sulfur compounds. It has a nauseating odor. Attempt was made to use limonite for gasoline purification. Almost complete removal of sulfide-sulfur was effected by limonite but last trace could not be removed by this. Thus purified gasoline could not stand Doctor's test. The treated gasoline was of rather dark color while the fresh one is of straw color. It may be mentioned here that fresh gasoline on long exposure to atmosphere became dark. Presently, the gasoline is being utilized as fuel in the power station. This is equivalent to twice its weight of coal.

During this experimentation with gasoline and limonite two important observations were made:

1) Hot gasoline dissolved considerable amount of sulfur from spent limonite and the crystalline sulfur could be obtained from rich gasoline on cooling.

2) The gasoline did not wet limonite and the treated limonite could be dried easily.

These stimulated to undertake the present work, the extraction of sulfur from spent limonite with gasoline and thereby to regenerate the spent limonite.

## Study of Gasoline

First, the study of gasoline was necessary to find out its suitability for the purpose. The characteristics of gasoline are presented in Table I.

TABLE I. CHARACTERISTICS OF CRUDE GASOLINE

| | | |
|---|---|---|
| 1. | Physical appearance | Straw colored liquid |
| 2. | Color on standing | Dark |
| 3. | pH | >7 (slightly alkaline) |
| 4. | Specific gravity | 0.79 at 20°C |
| 5. | Viscosity | 1.13°E at 20°C |
| 6. | Flash point | <20°C |
| 7. | Calorific value (gross) | 11000 cal./g. |
| 8. | Specific heat | 0.450 cal./g./°C at 37°C |
| 9. | Sulfide sulfur ($H_2S$) | 0.714g./l. |
| 10. | Sulfate | Trace |
| 11. | Total sulfur | 0.770g./l. |
| 12. | Phenol content | 3.5 g./l. |

**Fractionation of Gasoline.**—The composition of gasoline was not known. But it was reported³⁾

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* Present address: Natural Gas Fertilizer Factory, Fenchuganj, Sylhet, East Pakistan.

1) M. I. Ali, This Bulletin, 35, 878 (1962).

2) M. I. Ali, ibid., 35, 883 (1962).

3) A. H. Permantier, Private communication, 1957.

to contain 12.0% benzene. It is understood that higher aromatic hydrocarbons e. g. toluene or xylene are found to be good solvent for sulfur at an elevated temperature. So it was necessary to assess the availability of suitable fraction. The fractionation of gasoline was carried out by using a Vigreux model 800 mm. long fractionating column. The results are presented in Table II. Further, the fractionation of gasoline saturated with sulfur was also carried out, under similar conditions, for comparison.

TABLE II. FRACTIONATION OF CRUDE GASOLINE AND SULFUR IMPREGNATED GASOLINE

| Temp. °C | Distillate (%) by vol. | | Remark |
|---|---|---|---|
| | Crude gasoline | Sulfur saturated gasoline | |
| 40 | 0.6 | 0.2 | 1.0 l. samples were used for fractionation.<br>A thick dark residue was obtained in case of crude gasoline<br>Some carbon particles were also found therein.<br>In case of sulfur impregnated sample residual sulfur became dark plastic mass. |
| 50 | 3.2 | 1.0 | |
| 60 | 6.0 | 3.0 | |
| 70 | 10.0 | 4.8 | |
| 80 | 18.0 | 7.6 | |
| 90 | 29.2 | 18.0 | |
| 100 | 34.0 | 32.0 | |
| 110 | 40.0 | 40.0 | |
| 120 | 50.0 | 48.0 | |
| 130 | 55.4 | 54.0 | |
| 140 | 68.8 | 63.0 | |
| 150 | 72.0 | 74.0 | |
| 155 | 79.0 | 80.0 | |

From the close study of above results, gasoline fractions may be divided into 4 parts.

1) Fraction below 70°C……10.0% light fraction.
2) Fraction between 70～85°C……18.0% rich in benzene.
3) Fraction between 85～110°C……12.0% rich in toluene.
4) Fraction above 110°C……40.0% heavy fraction containing xylene and others.

The remaining 20.0% polymerized and decomposed at the distilling temperature. The light fraction and benzene fraction are obviously unsuitable for the purpose.

But it was observed that the residue was readily soluble in the light fraction. The solution was, of course, dark. However, for the practical purposes the gasoline fractions may be divided into two parts a) fraction below 85°C lighter fraction (LF) and b) the fraction above 85°C heavy fraction (HF). Heavy fraction was considered to be useful for the purpose. The above study revealed that there was not much influence of dissolved sulfur on fractionation except at the initial stage. The residue obtained was a plastic one, probably due to allotropic modification of sulfur. In view of the above observations, the solubility of sulfur in gasoline was studied only with the heavy fraction (above 85°C).

**Solubility of Sulfur in Gasoline.**—A sample of sulfur was recovered from the spent limonite by using gasoline. This was purified with LF gasoline and carbon disulfide. It was needle shaped crystalline sulfur of m. p. 114 (±0.5)°C. Optimum contact period for saturation of sulfur in gasoline and its solubility at different temperature were established. Eight minutes contact period at 70°C was found to be sufficient to reach saturation point. Therefore, 10 min. contact period was chosen for further solubility experiments at different temperature from 20 to 112°C. The results are presented in Table III (Fig 1). A depression of melting point of sulfur by 2°C was observed during these experiments. At 112°C excess sulfur formed a molten layer at the bottom.

The melting point of sulfur used in above experiments was 114°C. But in gasoline it melted at 112°C. The depression may be due to the presence of gasoline. Similar effect was also observed previously (melting point of crude

TABLE III. SOLUBILITY OF SULFUR IN GASOLINE WITH TEMPERATURE

| Temp. °C | Solubility | | Remark |
|---|---|---|---|
| | g./l. | % by wt. | |
| 20.0 | 6.5 | 0.77 | (a) At 112°C or above, molten sulfur formed dark layer at the bottom. |
| 50.0 | 22.7 | 2.62 | |
| 65.0 | 36.8 | 4.2 | |
| 80.0 | 51.6 | 5.74 | (b) True solubility at higher temperature above 85°C are different from the presented value. These require correction in the light of evaporation and change of composition of gasoline with the rise of temperature. Since the working temperature will be ultimately 85°C, these value are not so important. |
| 85.0 | 55.1 | 6.16 | |
| 90.0 | 62.9 | 6.96 | |
| 100.0 | 75.2 | 8.21 | |
| 110.0 | 90.0 | 9.68 | |
| 112.0 | Started melting | — | |

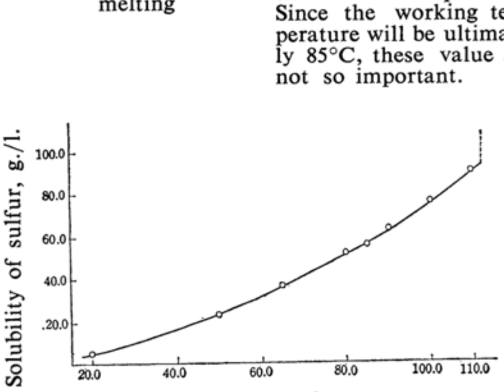

Fig. 1. Solubility of sulfur in gasoline with temperature.

recovered sulfur 111~112°C). However, the solubility of both the sulfur samples, purified in gasoline (LF) and carbon disulfide, were more or less the same. Solubility was expressed with respect to volume of gasoline at 16°C.

### Study of Limonites

A sample of spent limonite was collected from the heap and stored in well stoppered cans. The sample was analyzed and found to contain:

| | |
|---|---|
| Moisture | 13.5% |
| Sulfur (elem.) | 45.8% |
| Limonite etc. | Rest |

There are several varieties of limonite $Fe_2O_3 \cdot H_2O$ of which only $\alpha$ and $\gamma$ varieties are known[4) to be most active towards hydrogen sulfide. The structure and properties of these materials are susceptible to the climatic conditions. Temperature has much influence to that effect. Limonite looses its activity at high temperature. So, to retain the desired activity, the temperature is required to control within 40°C during normal use.

The main object of this work was to regenerate spent limonite and to recover sulfur as secondary product. A series of experiments were performed at different temperatures to find out the optimum regeneration temperature. The activity of each regenerated sample was studied (cf. Appendix I, part I). The results are presented in Table IV; Fig. 2.

It may be concluded from the above results that the activity of limonite decreases with increase of regeneration temperature and remarkably above 80°C. However, considering the solubility of sulfur in gasoline and activity loss of limonite with temperature, 85°C was chosen for further experiments. The loss of activity of limonite, at this regeneration temperature was about 10.0%. In view of above informations, a systematic study of limonite regeneration by gasoline was made with a batch process.

TABLE IV. ACTIVITY OF LIMONITE REGENERATED AT DIFFERENT TEMPERATURES

| Regeneration temp. °C | Activity % | Remark |
|---|---|---|
| 50 | 98.0 | The activity was calculated only on the basis of active iron (Fe) content in regenerated limonite according to the equation: $A$ (activity) $=\frac{X}{Y}\times 100$ where $X$=active iron (Fe) and $Y$=total iron present in the limonite. |
| 60 | 97.0 | |
| 70 | 95.0 | |
| 80 | 92.0 | |
| 90 | 85.0 | |
| 100 | 70.0 | |

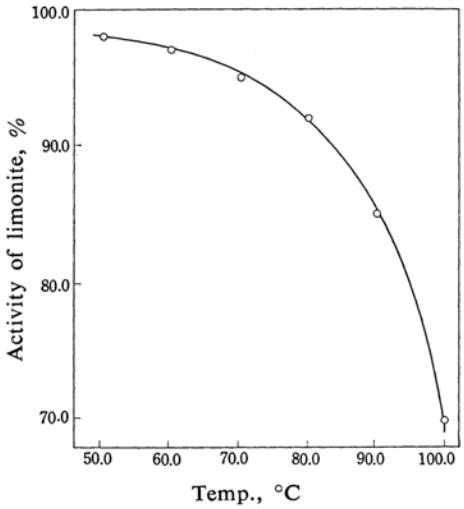

Fig. 2 Activity loss of limonite with temperature.

### Regeneration of Spent Limonite

**Batch Process.**—One hundred grams spent limonite was taken in a separating funnel containing 200 ml. gasoline (HF). The funnel with its content was immersed in a water bath at 85°C and agitated slowly. This was continued for 10 min. The solid-liquid mixture attained the same temperature of bath (85°C) within 3~4 min. Then, gasoline rich in sulfur was drained out and filtered at 85°C. Let this liquid be called "overflow". This was cooled down to 16°C and sulfur crystallized out was measured. Certain quantity of gasoline was retained by limonite mass and this liquid was termed "underflow".

Next, the mass in the separating funnel (including "underflow") was treated with another 200 ml. fresh gasoline in the same way as before. The batch leaching was continued until sulfur concentration in "overflow" became negligibly low.

The results of one set of experiments with 7th batch leaching are presented in Table V.

The progress of extraction falls too low with the increasing number of washings i.e. with the decrease of sulfur concentration in limonite. After sixth washing further treatment was almost useless. This fall of efficiency probably due to the fact that gasoline could not reach into the core of limonite particle to dissolve out sulfur therein entrapped. It is more clear from the extraction of sulfur (Table V), particularly extraction efficiencies there. It means

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4) R. H. Griffith and A. R. Morcom, *J. Chem. Soc.*, **1945**, 786.

TABLE V. EXTRACTION OF SULFUR FROM SPENT LIMONITE WITH GASOLINE

Temp., 85°C; Contact period, 10 min.

| No. of treatment | Gasoline / S. limonite (w/w) | "Overflow" gasoline recovery vol. % | Sulfur extraction wt. % | | Extraction efficiency % | | Remark |
|---|---|---|---|---|---|---|---|
| | | | Separately | Cumulative | Separately | Cumulative | |
| 1 | 1.68 | 66.5 | 14.8 | 14.8 | 92.5 | 92.5 | For each washing fresh gasoline was used. Gasoline distilled out with water content of limonite is neglected here. |
| 2 | 3.36 | 88.0 | 19.2 | 34.0 | 90.6 | 91.4 | |
| 3 | 5.04 | 95.5 | 20.1 | 54.1 | 87.6 | 90.0 | |
| 4 | 6.72 | 97.7 | 18.1 | 72.2 | 71.0 | 84.7 | |
| 5 | 8.40 | 98.7 | 9.8 | 82.0 | 42.0 | 73.1 | |
| 6 | 10.08 | 99.7 | 5.1 | 87.0 | 21.0 | 65.5 | |
| 7 | 11.76 | 99.7 | 1.9 | 88.9 | 8.0 | 56.6 | |

that a longer contact period is required. To produce the desired effect, it was thought that a modified procedure of continuous liquid flow at 85°C through the fixed bed of limonite would be suitable.

**Continuous Liquid Flow Processes.**—In view of the above observations, it was thought that a continuous liquid (gasoline) flow through the fixed bed of limonite may be useful. Due consideration was given to temperature condition, gasoline-limonite contact period, the quality of recovered sulfur and the by-products. Provision for direct feeding of crude gasoline was also kept in view.

It was thought that the distillation process would be suitable to effect both extraction of sulfur by reflux and separation of lighter fraction as distillate in the same process and equipments. To achieve this end, a modified equipment had been drawn up. It was also thought that the cyclic regenerative process would be a complementary measure against short supply of gasoline.

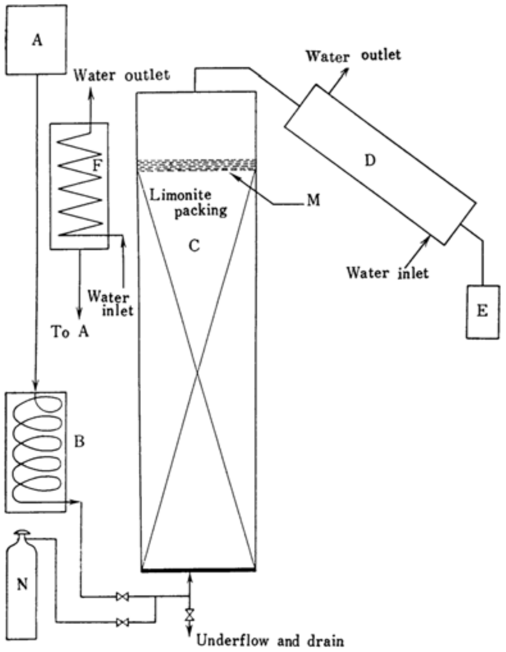

Fig. 3. Hot gasoline flow process.

| | |
|---|---|
| A | Gasoline reservior |
| B | Gasoline heater |
| C | Limonite column |
| D | Condenser |
| E | LF gasoline receiver |
| F | Overflow receiver |
| N | Nitrogen cylinder |
| M | Porous plug filter |

First attempt was made to separate lighter fraction of gasoline by distillation and utilize reflux gasoline for leaching the spent

TABLE VI. SOME PHYSICAL CONSTANTS OF GASOLINE AND LIMONITE AND THEIR USEFUL RELATIONS

1 Specific gravity of gasoline (HF): 0.84 at 16°C
2 Expansion of gasoline volume with temperature: 10.0% at 85°C on the vol. at 16°C
3 Specific heat of gasoline (HF): 0.545 cal./g./°C at 37°C
4 Latent heat of vaporization: 207 cal./g.
5 Packing density of spent limonite
  a) Loose packing: 725 g./l.
  b) Close packing: 820 g./l.
6 a) Free space in spent limonite packing (loose): 62.0%
  b) Free space in regenerated limonite packing (loose): 68.0%
7 Retention of gasoline
  a) HF by spent limonite: 37.0% (by vol.)
  b) HF by regenerated limonite: 38.0% (by vol.)
  c) LF by regenerated limonite: 36.0% (by vol.)
8 Reduction in volume of limonite on regeneration: 25.0%
9 Specific gravity of regenerated limonite: 0.52 (approx.)

limonite at the same time. But recovered sulfur was dirty while the heat loss was much. Moreover, the process was time-consuming.

In view of these observations it was further modified were hot gasoline at 85°C was passed through the fixed bed of limonite. By this "hot gasoline flow process" recovered sulfur was of 99.0% purity. The process is shown in Fig. 3. Arrangement was also made to dry up regenerated limonite by passing nitrogen gas through it and to recover lighter fraction of gasoline as distillate with steam. For smooth operation of the process, some physical constants of gasoline and limonite and their useful relations have been established. Those are presented in Table VI.

Having the above informations ready at hand to calculate the results under changing conditions, a systematic study of "hot gasoline flow process" was started. This was done

TABLE VII. RECOVERY OF SULFUR AND GASOLINE UNDER DIFFERENT CONDITIONS

| Experimental condition | Ratio (w/w) Gasoline / S. limonite | Gasoline recovered* |  | Sulfur recovered % | Water removed from limonite % | Remark |
|---|---|---|---|---|---|---|
|  |  | Rich % | Distillate % |  |  |  |
| I. Temp., 70°C; liquid velocity, 1.84 cm./min.; contact period, 10.9 min. | 2.0 | 89.2 | 10.8 | 18.9 | 66.0 | In all cases initially sulfur was slightly dirty but beyond gasoline - limonite ratio 2 it was bright. It tarnished on long standing in gasoline. Overall efficiency, 57.0% |
|  | 4.0 | 91.2 | 8.8 | 38.5 | 72.5 |  |
|  | 5.0 | 92.1 | 7.9 | 48.0 | 74.3 |  |
|  | 6.0 | 93.0 | 7.0 | 56.0 | — |  |
|  | 8.0 | 94.2 | 5.8 | 68.5 | 76.8 |  |
|  | 10.0 | 95.2 | 4.8 | 76.8 | — |  |
|  | 12.0 | 96.0 | 4.0 | 81.0 | 78.0 |  |
|  | 14.0 | 96.3 | 3.7 | 83.5 |  |  |
| II. Temp., 80°C; liquid velocity, 0.8 cm./min.; contact period, 24.7 min. | 3.0 | 89.2 | 10.8 | 40.8 | 74.5 | Overall efficiency, 61.5% |
|  | 5.0 | 91.5 | 8.5 | 63.5 | 77.7 |  |
|  | 6.5 | 92.8 | 7.2 | 75.0 | 78.8 |  |
|  | 8.4 | 94.2 | 5.8 | 83.5 | — |  |
|  | 10.5 | 95.2 | 4.8 | 88.1 | 79.9 |  |
|  | 12.5 | 95.9 | 4.1 | 90.0 | 80.0 |  |
| III. Temp, 84°C; liquid velocity, 2.3 cm./min.; contact period, 8.8 min. | 2.0 | — | — | — | 75.7 | Overall efficiency, 48.8% |
|  | 4.0 | 88.9 | 11.1 | 49.0 | 83.0 |  |
|  | 6.5 | 91.9 | 8.1 | 68.5 | 84.2 |  |
|  | 8.6 | 93.6 | 6.4 | 78.0 | 84.6 |  |
|  | 10.8 | 94.8 | 5.2 | 85.0 | — |  |
|  | 13.5 | 95.7 | 4.3 | 87.5 | 84.8 |  |
| IV. Temp., 86°C; liquid velocity, 0.64 cm./min.; contact period, 25.2 min. | 2.0 | — | — | — | 78.0 | Overall efficiency, 54.0% |
|  | 4.0 | 87.3 | 12.7 | 54.5 | 86.3 |  |
|  | 5.1 | 89.4 | 10.6 | 70.0 | 87.0 |  |
|  | 8.0 | 92.7 | 7.3 | 90.5 | 87.8 |  |
|  | 10.0 | 94.1 | 5.9 | 94.2 | — |  |
|  | 12.0 | 95.0 | 5.0 | 96.0 | 88.0 |  |
| V. Temp., 84°C; liquid velocity, 0.7 cm./min.; contact period, 27.8 min. | 2.0 | 84.0 | 16.0 | 26.3 | 74.0 | Overall efficiency, 49.2% |
|  | 4.0 | 89.5 | 10.5 | 53.5 | 80.0 |  |
|  | 5.6 | 91.4 | 8.6 | 74.4 | 81.5 |  |
|  | 7.3 | 92.8 | 7.2 | 84.0 | — |  |
|  | 10.0 | 94.6 | 5.4 | 91.0 | — |  |
|  | 13.0 | 95.8 | 4.2 | 93.5 | 82.5 |  |

* In the continuous liquid flow process "underflow" is assumed to be constant which is neglected in gasoline recovery calculations. This should be accounted only at the end of the experiment.

under varied operating conditions. The variables were (a) liquid velocity, (b) contact period of gasoline and (c) gasoline-limonite ratio, etc. The results of one set of experiments are presented in Table VII.

A close study of Table VII revealed that the best result was obtained under the conditions of experiment IV (temp. 86°C). The result of experiment V (temp. 84°C) was also almost equally good. These results are compared with that of batch process. The " hot gasoline flow process " was found to have some superiority over other two processes—" batch process " and " distillation process ", with respect to both sulfur recovery and its quality. Moreover, this was much advantageous from operational point of view.

It was interesting to note that the liquid flow and contact period have marked influence at the initial stage up to gasoline/spent limonite ratio 8. Beyond this ratio in all cases, the efficiency of extraction slowed down rapidly. From Table VII, it appears that sulfur extraction never reaches completion within reasonable gasoline/limonite ratio.

During these experiments, the percentage of gasoline escaped as distillate and the quantity of water eliminated from the system (i.e. from limonite) were also accounted.

However, a certain quantity of sulfur and water always remain in regenerated limonite. An average analysis of the same is presented in Table VIII for comparison with that of fresh limonite.

TABLE VIII. A COMPARATIVE STUDY OF FRESH AND REGENERATED LIMONITE

| Particular | Regenerated limonite* | Fresh limonite |
|---|---|---|
| 1. Humidity, % | 5~10 | 30~35 |
| 2. Sulfur content, % | 6~8 | Nil |
| 3. Iron content, % (dry basis) | 34.0 | 38.0 |
| 4. $H_2S$ absorption (Sulfur % on limonite) | 23.0 (against 26.% theor.) | 20.5 |
| 5. Efficiency**, % | 90.0 | Standard |

* This analysis was done before conditioning the regenerated limonite.

** This efficiency is on the basis of active iron content only. The presence of 6~8% sulfur reduces the iron content in the bulk and thereby lowers the efficiency. Finally, the overall efficiency of thus regenerated limonite stands at about 85%.

## Discussion

**Recovery of Sulfur.**—Impregnated gasoline on cooling yields crystalline sulfur of different shape and sizes depending upon mode of operation. Under ordinary condition, the crystals used to stick to the cooling surface and thereby retarded the heat transfer in the system. This would cause operation difficult. To make it much easier, direct quenching was tried and found to be satisfactory. The arrangement for the quenching process is shown by Fig. 4.

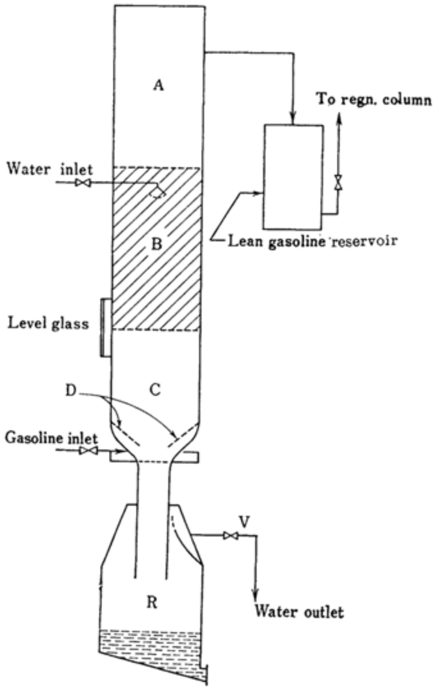

Fig. 4. Quenching column.
A Top zone
B Middle zone
C Bottom zone
D Distributor
R Receiver (sulfur settling)
V Control valve

The quenching apparatus consisted of a quenching column and a receiver for sulfur. Quenching column was a straight one, vertically placed on the receiver R. There were three distinct liquid phases in the column:

A Top zone: lean gasoline
B Middle zone: emulsion of gasoline-water.
C Bottom zone: clear water with bubbling gasoline and falling sulfur crystals.

The column was provided with a gasoline inlet at the bottom (at two points) under the distributor D, and exit at the top (zone A). Water was introduced at the top of middle zone. It was then allowed to flow down through the central clearance of the distributor, carrying sulfur to the receiver. Cryslals of sulfur settled down at the bottom of the receiver while water overflowed through the level controlling valve, V. A level glass would be necessary for this apparatus at the junction of zones B and C.

It is very important to maintain water level in the column and counter current flow ratio of gasoline and water rate. Gasoline flow at low pressure would stop if water flow is much higher. On the other hand increased gasoline flow would cause inefficient separation of sulfur. In short, the efficiency of the process is the function of (1) gasoline-water flow ratio, (2) gasoline-water contact period and (3) temperature of the liquids.

It was necessary to adjust all the variables for optimum condition. It has been observed that a streamline flow is required for effective cooling and separation of sufur. However, the working condition of a small column (30×2.5 cm. $\phi$) particularly at maximum flow rates is presented here:

a) Rich gasoline feed 500 ml./min. (445.0 g.)
b) Gasoline pressure 0.1 kg./cm$^2$
C) Gasoline-water contact period (in bottom zone) 3 sec.
d) Quenching water 579.0 ml
e) Temperatures of inlet and outlet water, −20°C and 50°C respectively
f) Temperatures of inlet and outlet gasoline, −83°C and 23°C respectively

Under this limiting condition the cooling efficiency was found to be 71.6%. A better efficiency could be achieved if the contact period is increased reasonably by increasing the column length and operating under higher gasoline pressure head. A modified column would help bettering stream-line flow of gosoline.

Crystalline sulfur accumulated in the receiver was of sizes arround 60 mesh. Some crystals used to agglomerate to form loose and soft lumps bound by emulsion film of gasoline while others moved freely in quenching water. On long standing bound gasoline in the lumps and emulsion film bubbled up to the surface. Thus settled sulfur became almost pure. It is interesting to note here that there was no appreciable oxidation of sulfur during quenching. Purification of recovered sulfur could be effected by steam treatment.

**Drying of Regenerated Limonite.**—It has been observed that a considerable quantity of gasoline was retained (underflow) in regenerated

TABLE IX. REMOVAL OF RETAINED GASOLINE BY NITROGEN

Regenerated limonite: 256.0 ml.
Gasoline to be removed: 97.0 ml.
Temperature: 60∼80°C

| Expt. | $N_2$/gasoline ratio (v/v) ×$10^2$ | Gasoline recovery wt. % | Remark |
|---|---|---|---|
| I. Gas flow 42 l./hr.; space velocity, 433 cc./cc./hr.; time, 83 min. | 2.18 | 50.0 | During drying process, the temperature was lowered as a precaution against activity loss of limonite. Too much lowering of temperature would cause drying inefficient. To maintain the temperature inlet nitrogen might be necessary to heat up occasionally. |
| | 2.93 | 74.5 | |
| | 3.8 | 86.0 | |
| | 4.7 | 93.0 | |
| | 5.65 | 96.5 | |
| II. Gas flow 12 l./hr.; space velocity, 124 cc./cc./hr.; time, 110 min. | 2.25 | 48.5 | |
| | 2.92 | 75.0 | |
| | 3.66 | 90.0 | |
| | 4.46 | 97·0 | |
| III. Gas flow 87.5 l./hr.; space velocity, 903 cc./cc./hr.; time, 43 min. | 2.21 | 49.5 | |
| | 3.0 | 70.5 | |
| | 4.05 | 81.0 | |
| | 5.0 | 87.5 | |
| | 6.0 | 91.0 | |
| IV. Gas flow 30 l./hr.; space velocity, 310 cc./cc./hr.; time, 114 min. | 2.23 | 49.0 | |
| | 2.92 | 75.0 | |
| | 3.71 | 88.0 | |
| | 4.54 | 96.5 | |
| | 5.52 | 99.0 | |

Note: (1) Nitrogen/gasoline ratio was calculated on the quantity of gas passed and total quantity of gasoline to be removed. (gas vol./liquid vol.)
(2) Average gas flow is indicated here.

limonite. Removal of this gasoline was important. Open air drying must not be permitted for its hazardous effect. It is inflamamble liquid and forms explosive mixture in air. It is also injurious for health. Its permissible limit[5] in U. S. A. is 1000∼1500 p. p. m. and U. S. S. R. is 55∼115 p. p. m. After careful consideration locally available nitrogen gas was the choice to be used for the purpose. A study of space velocity of carrier gas on gasoline removal system is presented in Table IX.

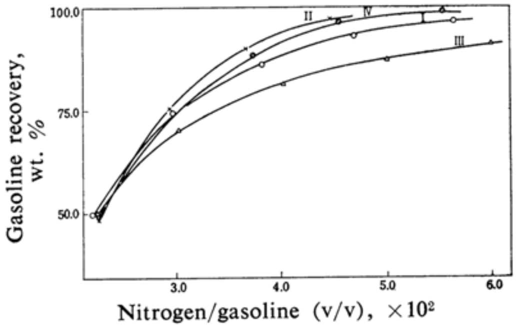

Fig. 5. Removal of retained gasoline by nitrogen.

I Space velocity 433 cc./cc./hr.
II Space velocity 124 cc./cc./hr.
III Space velocity 903 cc./cc./hr.
IV Space velocity 310 cc./cc./hr.

From above data and curves in Fig. 5 it is clear that gas flow and space velocity have bearing on the removal of gasoline from regenerated limonite. At higher space velocity, the removal of gasoline was relatively low (curve III), while at lower space velocity it was slow. An intermediate space velocity arround 310 cc./cc./hr. was found to be satisfactory.

Arrangements were also made to recover gasoline from the exit gas by oil washing and subsequent rich oil on distillation yields gasoline.

In conclusion, it may be said that the plant should be modified in the light of above observations by replacing the "overflow receiver" (F in Fig. 3) with a quenching column and adding one gasoline recovery unit.

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[5] V. J. Altieri, "Gas Analysis and Testing of Gaseous Materials", American Gas Assoc. Inc. (1945).