Standard Oil Company (Indiana)

INFORMATION DIVISION TRANSLATION T47-16

API-TOM Reel 213, Frames 6-11

Sludge Filtration Experiments with Different Solvents

In the continuation of earlier experiments, for filtering coal sludge mixed with solvents, a greater number of other solvents were investigated. A sludge from Chamber 804 of September 19, 1941 operating to make naphtha-fuel oil, was used, which was mixed with the solvent concerned in the ratio of 1:2 and was stirred at an increased temperature for one hour. The subsequent filtration was carried out as customary (see report of Dr. Lemme) and the products which came out analyzed. The experiments showed that the different solvents affect the sludge quite differently and markedly alter it, in part. A correlation between chemical constitution of the solvent and its behavior could not, however, be verified. Worthy of note is that individual solvents act differently in the cold than in the warmth. In order to obtain certain criteria for the course of the filtration, the preliminary experiments were carried out in reagent glass and the solvent ability, rate of filtration and the appearance of the collected products were observed. Thereby, are already shown great differences in the behavior of individual solvents. The filtration times of the main experiments, arranged according to rate, in the individual solvents are the following:

acetyl-chloride	3	seconds
heavy naphtha	6	"
chloral	13	"
solvent naphtha("schwerbenzol")-	21	"
styrene	28	"
dioxan	67	"
nitrobenzene	67	"
acetophenone	107	"
chloroform	185	"
cyclohexanol	186	"
ethylene chloride	218	"
aniline	285	"
pyridine	312	"
phenol	903*	"
acetone	842	"

To be emphasized is the good filtration time of acetyl chloride which is remarkable in other ways yet to be considered further in detail, as well as the poor filtration time of chloroform, ethylene chloride and acetone which must be further investigated, since its behavior was quite different in the test tube. From the introduced oil and asphalt amounts 80-99% was found in the filtrate whereby the good oil solvents stand at the top, chloroform, acetone

*(Tr.: apparently should read 803)

and others, to be sure, fall out. The introduced asphalt was found to 16-92% in the filtrate, the remainder was partly in the filter residue and in part disappeared. In the latter respect, acetyl chloride and acetone were outstanding, in that almost half of the asphalt was not found again.

On the basis of the above-described results a semi-technical experiment was recommended with the solvents - heavy naphtha, solvent naphtha, acetyl chloride and acetone. Further specific experiments were instituted with these solvents, in part in mixture and with modified amounts and reaction conditions.

Experimental Conditions

In support of earlier filtration experiments one part of sludge from chamber 804 of September 19, 1941, operating to make naphtha-fuel oil with 22.0% solid and 78.0% oil plus asphalt (20.4% asphalt) was stirred with two parts of solvent in a 3-necked flask with a stirrer, thermometer and reflux cooler for one hour at a temperature which lies 5-10° below the boiling point of the particular solvent but not over 140°. In this connection 100 g. of the blend was filtered through a steam-heated 7-cm. diameter vacuum filter at the stirring temperature and the filtration time measured. Filtrate and filter residue were weighed and analyzed.

Oil-plus asphalt, asphalt and solid were determined.

Results

1) Filtration times

acetyl chloride	3 seconds
heavy naphtha	8 "
chloral	13 "
solvent naphtha(schwerbenzol)	21 "
styrene	28 "
dioxane	67 "
nitrobenzene	67 "
acetophenone	107 "
chloroform	185 "
cyclohexanol	186 "
ethylene chloride	218 "
aniline	285 "
pyridine	312 "
phenol	803 "
acetone	842 "

2) Oil plus Asphalt in the Filtrate Calculated on the Charge

solvent naphtha	98.8%
phenol	96.8%
styrene	92.4%
aniline	90.5%

acetyl chloride	90.3%
heavy naphtha	85.5%
chloral	84.0%
acetophenone	82.3%
ethylene chloride	81.6%
pyridine	80.8%
dioxane	76.2%
nitrobenzene	74.5%
cyclohexanol	73.1%
chloroform	73.1%
acetone	70.0%

3. Asphalt in the filtrate oil calculated on charge.

phenol	92.5%
heavy naphtha	90.5%
aniline	90.5%
solvent naphtha	77.3%
pyridine	75.4%
dioxane	73.6%
ethylene chloride	70.0%
nitrobenzene	57.0%
styrene	56.6%
acetyl chloride	50.8%
chloral	23.8%
chloroform	17.8%
cyclohexanol	17.8%
acetone	17.4%
acetophenone	16.5%

4) Asphalt in the oil of the filter residue.

acetyl chloride	0%
phenol	6.3%
heavy naphtha	8.9%
aniline	9.5%
dioxane	10.1%
pyridine	21.1%
ethylene chloride	21.3%
solvent naphtha	21.4%
styrene	35.8%
nitrobenzene	37.5%
acetone	41.2%
cyclohexanol	45.7%
chloroform	62.8%
chloral	64.9%
acetophenone	74.7%

5) Asphalt loss

acetyl chloride	49.0%
acetone	41.4%
cyclohexanol	36.5%
chloroform	19.4%

dioxane	16.3%
chloral	11.3%
acetophenone	8.8%
ethylene chloride	8.7%
styrene	7.6%
nitrobenzene	5.5%
solvent naphtha	1.3%
phenol	1.2%
heavy naphtha	0.6%
aniline	0 %
pyridine	0 %

6) Filtration residue

acetyl chloride	0.6%
styrene	9.2%
aniline	10.0%
heavy naphtha	10.7%
nitrobenzene	12.2%
chloroform	13.5%
cyclohexanol	13.6%
acetophenone	13.8%
pyridine	14.0%
ethylene chloride	14.0%
solvent naphtha	14.3%
phenol	14.8%
dioxane	20.2%
chloral	24.6%
acetone	25.2%

Concerning the filtration time, acetyl chloride, heavy naphtha, chloral and solvent naphtha are outstandingly good; styrene, dioxane, nitrobenzene and acetophenone yet completely passable; noteworthy is the poor time of chloroform, ethylene chloride, pyridine and acetone. The latter filtered very well in the test tube experiment (without long heating) and left behind a coarse-grained precipitate.

Concerning the oil plus asphalt value in the filter oil the poor yield from acetone and chloroform is again to be mentioned, while solvent naphtha, phenol, styrene, aniline and acetyl chloride gave good yields.

The asphalt values of the filter oil show great differences in the individual solvents. Outstanding are the low values in acetophenone, acetone, cyclohexanol, chloroform and chloral and also in acetyl chloride, styrene and nitrobenzene. This is at this time consistent with the values of the asphalt losses which are fairly high in the cases of acetyl chloride, acetone, cyclohexanol and chloroform.

The amounts of filter residue are very small with acetyl chloride, very high with acetone, chloral and dioxane. Otherwise they move about the normal value (10-15 g.)

Conclusion:

Of the solvents greatly used technically, heavy naphtha, solvent naphtha, acetyl chloride and acetone are suitable on the basis of the experimental results for the semi-technical sludge filtration experiments.

/s/ Leonhardt
Lemme

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Checked Feb. 18, 1947 - CCM

"Abschlammfiltrations Versuche mit verschiedenen Losungsmitteln".
I.G. Farbenindustrie A.G.
Hochdruckversuche
October 21, 1941
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Standard Oil Company (Indiana)

INFORMATION DIVISION TRANSLATION T47-17

API-TCC Reel 215, Frames 12-15
Sludge Filtration Experiments with different Aldehydes.

Summary and Conclusion

In continuation of earlier sludge (from operation to make naphtha-middle oil and naphtha-heating oil) filtration experiments in the application of furfural as diluent in the ratio 1:2, additional aldehydes were tested under like conditions. Measured on filtration time, arranged according to speed, yielded the following picture:

1.)	i-butyraldehyde	5 or 10 sec.
2.)	n-butyraldehyde	11 or 16 sec.
3.)	crotonaldehyde	12 or 14 sec.
4.)	furfural	46 or 72 sec.

Those out of bounds are: formaldehyde (552 or 618 sec.); propion-aldehyde (38 or 300 sec.); benzaldehyde (45 or 394 sec.); acrolein (5 or 23 sec. however in one case (Naphtha-Middle Oil) shortly after filtration, polymerization to brittle solid substance); acetaldehyde (showed a separation into two phases, whereby the total solid constituents were in the lower phase). For comparison sludge was diluted with middle oil 1:2 and filtered (filter time 300 sec.).

The analytical investigation showed for all aldehydes that 75-95% of the introduced sludge-oil and 47-80% of the asphalt are contained in the filtrate. The remaining amounts of asphalt are found in the residue. With furfural there comes an excess of 28% (new formation from oil). In order to be able to compare between the individual values of the asphalt analyses and coke tests, there were made, at present, asphalt analyses and coke tests of the filtrate oil and the residue of the experiments with i-butyraldehyde, n-butyraldehyde, furfural and crotonaldehyde, and were compared with the values of the unchanged sludge.

On the basis of previous investigations, a semi-technical experiment with i-butyraldehyde is recommended.

Furfural in mixture with other solvents will be further tested.

According to earlier report, the filtration experiments of sludge from chamber 804 (August - October 1941; Upper Silesia coal) with furfural came out very well. They were continued, therefore, with a greater number of other aldehydes. Applied were sludges from the operation to make naphtha-middle oil and naphtha-fuel oil.

Experimental Conditions

The experimental conditions were the same as with furfural, i.e. one part of sludge was mixed with 2 parts of aldehyde and stirred one hour

at a temperature which lies 5-10° below the boiling point of the concerned aldehyde but does not exceed 140°C. After stirring, 100g. of the mixture was filtered through a 7 cm. diameter suction filter heated to the stirring temperature. Measured were the filtration times, filtrate and amounts of filter residue. The solvent - free resulting products were analyzed.

Results

Of the aldehydes applied, formaldehyde showed such a high filtration time that further investigations were spared. Acetaldehyde effected a separation into 2 phases of which the lower contained the total solids blended with asphalt. The upper phase was practically free of solids and can be separated without a filter. Also here, further investigations were not carried out. Propionaldehyde showed throughout in the operation to make naphtha-fuel oil a tolerable filter time value of 38 sec.; while it gave 300 sec. in the operating to make naphtha-middle oil, a value which is practically the same as in dilution with middle oil. Further investigations therefore, were not discussed. Acrolein indeed showed throughout favorable filtration times, but the filtrate from the experiment of the naphtha-middle oil operating procedure polymerized spontaneously into a solid brittle substance. From this view point also the possibility of spontaneous polymerization is usually indicated. From this basis exhaustive discussion is also renounced. On account of the great differences of the filtration times, benzaldehyde is also eliminated.

The remaining aldehydes (i-butyl, n-butyl, crotonaldehyde and furfural) were considered under the following points:

Method of Operation	Naphtha-Fuel Oil	Naphtha Middle Oil
I.) Filtration Time		
i-butyraldehyde	5 sec.	10 sec.
n-butyraldehyde	11 sec.	16 sec.
crotonaldehyde	12 sec.	14 sec.
furfural	45 sec.	72 sec.
2.) Filtrate oil in % of Charge.		
i-butyraldehyde	90.5%	94.2%
n-butyraldehyde	88.2%	89.0%
crotonaldehyde	84.4%	92.0%
furfural	79.7%	87.7%
3.) Asphalt in the filtrate oil in % of charge		
i-butyraldehyde	80.8%	69.7%
n-butyraldehyde	71.6%	70.7%
crotonaldehyde	66.2%	72.8%
furfural	65.0%	60.8%

4.) Filter residue

i-butyraldehyde	14.2 g.	8.8 g.
n-butyraldehyde	16.8 g.	9.8 g.
crotonaldehyde	11.7 g.	9.4 g.
furfural	13.3 g.	10.5 g.

5.) Asphalt in the oil of the residue in % of the Charge

i-butyraldehyde	13.5%	32.1%
n-butyraldehyde	25.7%	25.5%
crotonaldehyde	29.0%	25.5%
furfural	62.6%	37.5%

Conclusion

From the experimental results come forth that especially i-butyraldehyde is suitable for the working up of coal sludges. Furfural, which is technically abundant, used as extraction solvent should, on account of its poor filtration properties, be further tested in mixture with other solvents.

Signed/Leonhardt/Lemme

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Checked by C. C. M. February 19, 1947
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