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High Pressure Experiments
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INFLUENCING THE RATE OF FILTRATION OF BITUMINOUS HYDROGENATION PRODUCTS.

It has already been observed with most coals that if the prehydrogenation has been permitted to go too far, the filtration rate is reduced by a conversion of primary bitumens. In addition at the beginning of operations, when hydrogenation HOLD had to be used for the thickening of the pasting oil, because of a lack of a return HOLD from the production, the conversion products of the coal were unfilterable, and normal operations could not be started for several days. The nearly ash and carbon-free heavy oil let down obtained on continued hydrogenation of primary bitumens filters much more poorly after being diluted with pasting oil than the original products.

Results of a few tests shown below confirm that even if hydrogenation let-downs are added later to solutions of coal, the rate of filtration drops to a very small proportion.

A conversion product of upper Silesian coal was used with a pasting proportion of 1 : 1 and diluted with 25 percent of pasting oil, 200 - 300°C, from converter 470; increasing the amounts, from 1 to 15 percent, of the Scholven centrifuge residue were added to the above mixture and the rate of filtration through a suction filter of about 40 sq. centimeter filtering surface was determined after an extensive mixing for three hours at the filtration temperature of 150°C. The filtration times for 100 g of the thinned out material were as follows:

Additions	Filtration time Min.	Sec.	Yield sq. meter/h
Original Mixture	-	54.8	1,640 kg
+ 1.0 percent centrifuge residue	1	17.6	1,150 "
" + 4 percent	1	57.2	766 "
" + 8 percent	2	11.4	683 "
" + 15 percent	2	42.6	553"

Plotting the time of filtration of the diluted coal conversion products in seconds against the percentage additions of the hydrogenation residues will give the curve shown in the appendix.

Additional tests were made to determine whether the nature of the ash affected the rate of filtration. To do this a solid-free oil was obtained by filtration from the above centrifuge residue as well as from the

catchpot heavy oil, and to this different amounts of the finest powdered silica were added to replace an acid ash, and chalk as a basic constituent, and after stirring for three hours at about 140°C, the time of filtration/100 g of the mixture was determined.

The filtration times were determined for 100 g filtrate from a suction filter of about 40 sq. centimeter.

Original Material + Additions	Filtration time in seconds	Ash in the filtrate	Remarks
Catch-pot heavy oil, Ash-free, from converter 451, by itself	108	0.03	
" + 1 percent SiO_2	145	0.02	
" + 1 " $CaCO_3$	19	Traces	About 6 fold productivity
Catch-pot heavy oil, Ash-free, from Converter 451, by itself	125	0.006	
" + 10 percent SiO_2	105	Traces	
" + 10 percent $CaCO_3$	20	0.02	About 6 fold productivity
Centrifuge oil II Ash-free, by itself	13	0.02	
" + 1 percent SiO_2	77	0.02	
" + 1 percent $CaCO_3$	16	0.02	

The table shows that SiO_2 and $CaCO_3$ have entirely different effects upon the rate of filtration of the ash-free (filtered) heavy hydrogenation oils. SiO_2 added within the limits of 1 - 10 percent affects the filtration time not very much, mostly in the negative way; while the addition of calcium carbonate favors very favorably the rate of filtration; with catch-pot heavy oils the rate of filtration is six times as great when with 1 - 10 percent $CaCO_3$; if adding to the solid-free centrifuge oil II, the addition of lime results in no improvement of the already high rate of filtration, but even in this case the addition of $CaCO_3$ has about a five times more favorable effect, than the addition of the same amount of SiO_2. The hydrogenation middle oils are practically not affected by the two additions.

The occasionally extraordinary improvement of the filtration by the addition of $CaCO_3$ appears to be based upon a flocculation of the high molecular weight (acid) sludge materials by the alkaline calcium carbonate. This is confirmed by the increase in the filtration residue which amounts to 46 percent with a 1 percent addition, and to only 11.8 percent with a 10 percent addition in case of the catch-pot heavy oils; in the case of the centrifuge oil the corresponding numbers

are 80 percent and 12.5 percent; i.e. with smaller amounts of chalk, more of the acid sludge materials are precipitated than of with 10 times greater amounts of chalk, which again proves that precipitated calcium carbonate acts better than the crystalline calcium carbonate, e.g. ground marble. With the same additions of ground marble the filtration time is somewhat higher and the addition to the filtration residue somewhat lower.

The effect of $CaCO_3$ upon the ash-containing filtration let-downs is unfortunately not as well noticeable, because the filtration time is so very high, and even if there is an improvement of about 300 percent by the addition of 1 percent $CaCO_3$, and a further improvement of 25 - 30 percent when the amount of the $CaCO_3$ addition is increased from 1 percent to 10 percent, still the total filtration time remains unsatisfactory. The tests are being continued.

/s/ Pfirrmann
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Effect of Centrifuge Residue on the Time of Filtration of Ash-Free Solutions of Bitumens