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UNITED STATES
DEPARTMENT OF THE INTERIOR
BUREAU OF MINES
OFFICE OF SYNTHETIC LIQUID FUEL
LOUISIANA, MISSOURI

From Dr. Pier's file

T-432

High Pressure Experiments
Ludwigshafen, 558

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January 7, 1948

DIFFICULTIES ENCOUNTERED IN THE FOAMING METHOD OF
SYNTHESIS OF HYDROCARBONS, AND THEIR OVERCOMING
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February 18, 1943, M1/Fu

No fundamental difficulties have ever been met in the foam process. It may well be stated, therefore, that there remain no important unsolved problems in the process. Difficulties of the second order of magnitude have however been encountered, and they have caused interruptions in the 1.5 cbm reactor. They will be discussed here, and means used for overcoming them will be explained.

1). Leaks in Stuffing Boxes and Valves.

The contents of the reactor sump must be kept in circulation by pumping to cool them and to degasify them. A centrifugal pump is used for the purpose. Even if it were at all possible to set the pump drive directly inside the high pressure space, thus avoiding the need for a stuffing box for the shaft, it would still be preferable for constructional reasons and for

control to do it as it is now done and to lead the shaft outside through a stuffing box. Such stuffing boxes have already been successfully used in pressured spaces.

The problem here is, however, made more difficult by the necessity of avoiding leaks in a sump space which contains a suspension of iron, iron oxide, iron carbonate and iron carbide. The suspension has a pronounced grinding effect when it enters the space of the shaft stuffing box. Deep enough grooves are quickly formed on the shaft to make it unusable. To overcome this difficulty about 40 kg of grease is forced every day into the stuffing box space, and this prevents the converter sump liquid from entering it.

The Burgmann packing, impregnated with oil and graphite has been found satisfactory.

The stuffing box packing becomes gradually used up, and it must be tightened. When this is no longer possible, a reserve parallel pump, with valves closed when not in use, is connected up, the first pump is disconnected and taken out for repairs or repacking of the stuffing box. Several valves are required for this change, with a large bore, so as not to narrow down the sump outlet. This naturally will necessitate having a few more stuffing boxes for the valve stems.

It is no longer difficult to-day to install valve stuffing boxes in pressured vessels. Never the less, the failure of a Klinger valve caused by the peculiar properties of the sump was the cause of a stall fire in the fall of 1942. The stuffing box packing which evidently had been made too weak, sprung a leak. Steps taken immediately were to no avail, because the sump material inside the packing produced channels in the course of a few minutes, began spattering outside, as it always does in cases of this kind, and caught fire because of the presence of pyrophoric iron in the sump material.

The Klinger valves have been replaced with slide valves of an American design which have so far been found satisfactory.

2). Settling of the Catalyst in the Reactors.

The tendency of the catalyst in the sump to stick to the wall surface acts as another source of trouble. This behavior has been discussed in detail on 11/28/42 in a report entitled "Deposition of Catalysts on the Reactors Walls in the Foaming Process". It becomes manifest by the formation of gradually thickening scaly deposits of catalyst on the inside wall of a reactor. A similar phenomenon can be observed in the degasifying pot, and in short in any place where

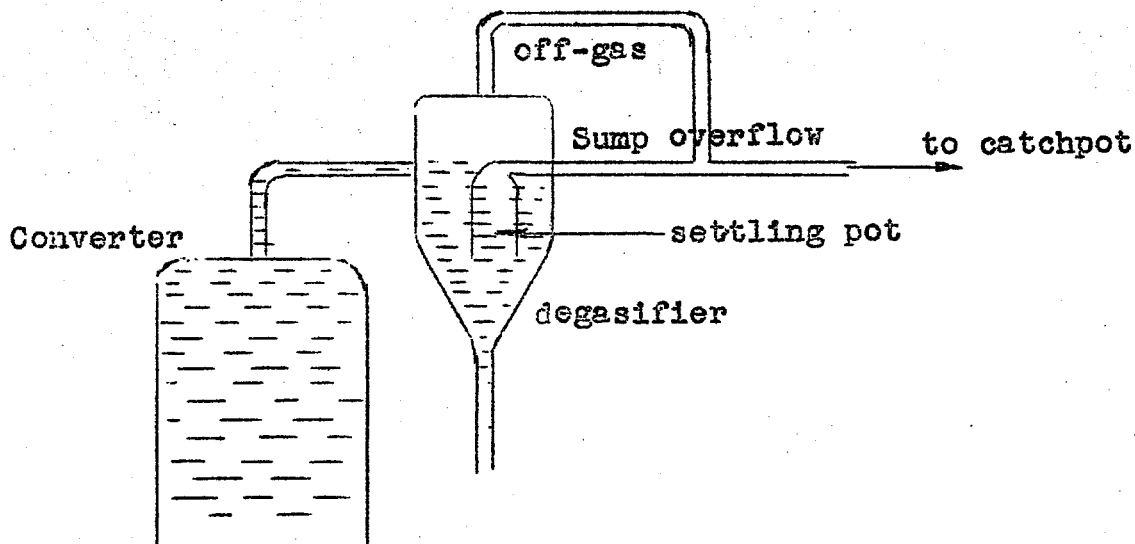
the liquid is not in a violent motion. This deposit is not formed by a sedimentation caused by gravity, but by the adhesive action of some difficultly soluble substance formed in the sump, the nature of which can at present only be suspected.

The formation of scales gradually removes increasing amounts of the catalyst from the reaction space and noticeably reduces the reaction space, and the trouble was finally overcome by finding mechanical means of preventing the formation of the deposit. A kind of scraper was introduced into the reactor, a frame structure which can be rotated by means of a shaft in the cylinder axis of the reactor, and which scrapes the reactor wall and removes the catalyst deposit. The scraper shaft is carried out of the furnace in its upper part through a stuffing box and is connected to a hand operated conical drive. The scraper is operated only occasionally, say every four hours, so as not to put unnecessary strains on the stuffing box. It operates very easily, and has disclosed the surprising fact, that the carbon dioxide content of the end gas, which is a measure of the gas conversion, is increased after each stirring, e.g. from 30 to 32 percent, if it was low before. We can not explain this fact by scraping off a corresponding amount of the catalyst

from the reactor walls, because a sufficient amount of the catalyst could not have deposited since the previous scraping. One is rather forced to assume, that there probably exists a loose sedimentation of the catalyst on the bottom of the converter, which is again brought into circulation by the scraper. The stuffing box on the scraper shaft must be tightened from time to time, just like all the others.

3. Sticking of the Catalyst in the Gas Filled Part of the Degasifying Pot.

The sump material entering the degasifier from the reactor enters below the liquid level to avoid spattering on the upper wall. Never the less, because of the short distance of the level of the liquid in the sump from the cover, spray from the latter reached the cover, and deposits were formed on it for the same reasons as on the reactor walls, and kept growing.



This made itself primarily inconvenient by reducing the upper gas outlet. The process was aided by the thermocouple tube, located centrally in the gas outlet tube and which facilitated its plugging up. If the gas outlet was completely closed, the gas pressed the liquid level down to the bottom edge of the settling pot, to discharge through the latter into the catchpot. The settling pot became empty and its contents were pressed into the catchpot.

This resulted in important difficulties. Small variations in the amount of liquid in the sump, caused by slight variations in the gas supply, emptied the piece of tubing on the bottom of the settling pot because of the absence of appreciable reserves in it. Relatively dry deposits of the catalyst formed in the settling pot, occasionally chipped off and reached the pump, produced abrasions in it and necessitated a shut-down.

When all these disturbances were first noted, the reason for them were sought elsewhere, no one thought of the possibility of plugging up, because the converter had operated previously for three months without any plugging up. Finally, however, the failure of the gas outlet tube remained as the only possible explanation, and a dismantling proved it to be the correct one.

The difficulty was overcome by first of all removing the thermocouple centrally located in the gas outlet tube, which favored the deposition of the catalyst. The reactor has operated now for four months without any plugging up.

Additional precautions used in the 14 cm reactor then under construction: the liquid from the sump was made to enter the degasifying pot underneath the level of the liquid, as before, and 1 m distance was maintained between the degasifier cover and the level of the liquid. It was expected, that this would prevent the spattering of the liquid upward.

Summary

Large scale experimental work on the foaming process has led to no difficulties which might be considered fundamental. Any troubles encountered were caused by stuffing boxes, chiefly of rapidly rotating shafts, and by the sticking of the catalyst to the wall surfaces in the sump and the gas space. The difficulties could be overcome by relatively simple measures.

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