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MODIFICATION OF A CATALYST HYDROCRACKING REACTOR TO PERMIT COAL-BITUMEN SLURRY PROCESSING AND A COMPARISON OF OPERATING CHARACTERISTICS.

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INTRODUCTION

The objective of the following work was to demonstrate that a slurry pump operation can be used in a bench scale flow system for the hydrocracking of Athabasca bitumen. One project at the Energy Research Laboratories located in Ottawa involves the use of fine mesh coal as a Catalyst support or as a disposable catalyst for the hydrocracking of crude oils. To implement this project the present bench scale fixed bed flow system had to be modified in such a way as to permit a slurry pump operation.

THE OPERATIONAL PROBLEM

It had been noted during fixed bed flow system runs with coal catalysts used that coal in a large amount mixed with bitumen congeals and forms slugs in the system. This plugs various product lines and does not permit the run to continue. The coal, once deposited in a small amount, tends to "pick-up" any other coal particles passing through the system and eventually cause a large enough build-up of coal to stop the system during the experiment. This problem could be over-come by creating a back-pressure in the system and blowing the plug through, but the undependability of this procedure led to the slurry pump approach. During a fixed bed flow system operation the catalyst is placed in the reactor, in a large amount, in a chamber with bitumen passing through in the presence of hydrogen. In a slurry pump system, a fine mesh coal is mixed with bitumen to the desired ratio in a feed hopper until a homogeneous mix is attained, then fed through a reactor in the presence of hydrogen to obtain the same product. This results in reduced maintenance time between catalyst life runs; prevention of catalyst slugging; and because of design, coal build-up become infrequent.

The bench scale fixed bed flow system was fed by a positive displacement proportioning pump. A slurry application of this pump would score its highly polished internal cylinder surface and reduce its metering accuracy through time. To circumvent this problem a controlled volume reciprocating positive displacement pump was used. Basically, the pump consists of a drive unit, a plunger, and a displacement chamber or liquid end in which the plunger reciprocates. Thus the coal slurry rises vertically through the pump and does not come in contact with the accurately machined and highly polished surfaces of the pump.

APPLICATION OF THE POSITIVE DISPLACEMENT PUMP

The experimental work was undertaken in a modified bench-scale flow system using a "Milroyal" controlled volume reciprocating positive displacement pump installed directly below the reactor as shown in Figure 1. A feed hopper was then installed close to the pump but higher than the pump head in order to provide a natural gravity feed flow, through the pump head. A heated and insulated hopper line was then connected between the hopper and the inlet end of the slurry pump. For reasons outlined in the pump instruction manual (2) this line was three times larger in diameter than the lines leading off the pump outlet.

The feed hopper is a stainless steel rocket shaped container. A four propellor air mixer is used as a continuous mixing device to mix the feed and slurry initially and then keep a homogeneous mix throughout the run. A valve was placed on the hopper line close to the bottom of the hopper to keep all the feed in the hopper until a homogeneous mix is attained. The hopper is heated to 80^{o}C during runs and controlled by an independent thermocouple and carefully monitered at all times.

The slurry pump is equipped with ball and check valves located above and below the pump head which operate in sequence, allow feed into the pump head chamber, closing the inlet balls; then the outlet balls open permitting the feed to pass into the feed lines. A teflon packing is used in the stuffing box located outside the feed chamber of the pump. This must be periodically lubricated during runs to prolong the life of the various moving parts of the pump head.

The feed line is a heated and insulated line connected from the outlet end of the pump head to the base or inlet end of the reactor. On this line, close to the pump head, a 5000 lb. rupture disc (Fig. 1) is located to protect pressure build-up in the feed line from damaging or blowing back through the pump head. The feed line is also hooked up in such a way that the pump can be started in atmosphere to make sure that the pump is pumping, then by closing and opening two high pressure valves, and placing a hydrogen pressure on the pump head, the pump then is ready for operation. Also located on this line close to the reactor end is a reactor drain valve so that feed trapped in the reactor after running time can be removed. This is helpful when a reactor sample is needed to determine what reaction is taking place in the reactor during runs.

The reactor used is a three heater reactor, the same as the reactor used in fixed bed flow system runs (1). To provide easier assembly and more working room around the reactor the feed inlet and outlet plug ends were reversed without reversing the reactor. This permitted the hydrogen line to be connected to the feed line directly beneath the reactor without having the thermocouple well and thermocouple hindering the operator's work. This also permitted the feed line to be connected to the base of the reactor without an unnecessary bend in the feed line. The hydrogen and feed meet in the tee connector at the base of the reactor and bubble up together through the reactor as in a fixed bed bench scale flow system. (1) The thermocouple then, also is in an inverted position at the top, or outlet end of the reactor, giving it more freedom to do profiles. The flow of the product from the outlet end of the reactor to the sample vessels is unchanged from the fixed bed bench scale flow system (1).

A slurry wash out system was installed immediately below the hopper valve (Fig. 1) complete with its own valve, to the hopper line. This enabled the hopper line, the pump ball valves and pump chamber, and part of the feed line to be cleaned out of slurry between runs so that slurry build-up on the ball valves and in various lines could not occur.

The slurry wash out system consisted of a pyrex burette placed above the feed hopper to provide a natural gravity flow with tygon tubing running from the bottom of the burette to the hopper valve. A light gas oil was used as a wash out fluid so that the wash out system lines did not have to be heated. All other facets of the operation remained the same as in a fixed bed bench-scale flow system (1).

EXPERIMENTAL RESULTS AND DISCUSSION

During the first run and subsequent runs of the slurry pump various problems were encountered that were particular to the properties of slurried bitumen . It was noted first off that heated lines had to be controlled closly, the temperature neithor rising, or falling. If the temperature were too low, the flow of bitumen would cease which is common with bitumen but if the temperature were too high the lines would coke up much faster than with just pure bitumen. This was especially noticeable in the hopper line between the hopper and the inlet end of the pump head.

It was also noted that the slurry must be moving all the time whether agitating in the hopper or flowing through the lines. Therefore it is inadvisable to leave slurry in any of the various lines during maintenance time. The slurry will sink to the bottom of the line and form into a sludge-like consistantcy and will not move and must be hand cleaned. This also is true in the feed hopper. Therefore once a particular slurry has been added to bitumen in the hopper, the hopper must always be kept heated at the correct temperature with the mixer kept on and then emptied as much as possible at the end of the day. For this reason also the slurry wash out system was developted to keep the ball valves in the pump clear of slurry during shut down times. It is also advisable, when constructing the feed and hopper lines to make them straight as possible with little or no bends. Slurry tends to build up at any point where it gets a chance to settle.

It is advisable to mix the coal with the bituman in the hopper and then leave it with the mixer running for one hour to obtain a homogenious mix. The hopper has a 3000 ml. capacity but it is advisable to fill the hopper only one third full so that a minimum amount of coal slurry is present and less will be left in the hopper between runs.

It is also advisable to have hydrogen flowing through the reactor by the time the reactor reaches 250^{o}C and feed flowing through the lines and the reactor by the time that the reactor reaches 350^{o}C. Calibration experiments were done through the pump into atmosphere and through the pump and reactor into sample vessels at 2000 PSI hydrogen atmosphere. Samples were taken at various percent stroke length on the pump and at various percent motor speed on the pump motor. It was found that a direct relationship occured between the two atmosphere's (Fig. 2). In each case weight yields versus time were measured.

CONCLUSION

The differences in a fixed bed bench scale flow system and a slurry pump system are many, each having their particular use in catalyst evaluation.

In a slurry pump system very little,if any Coke was present in the reactor after runs. The reasons for this, it is felt, is two-fold. one - there is less coal present in the reactor at any one time and therefore build up is minimal. Two - The coal does not tend to slug and plug lines because it is constantly moving through the system and build-up does not occur. Also as an added plus there seems to be less temperature controlling problems during runs because of less slugging in the lines. The hydrogen flow does not seem to be affected as much.

It was noted that the pump mounted directly beneath the reactor and as close as possible to the bottom of the reactor with no bends in piping is extremly desirable because a very close direct force is applied to the feed from the pump giving very little time or space for the coal to settle back down on the pump head. The slurry cannot settle anywhere it must go through the reactor.

The feed mixing became a very simple problem once it was realized that the hopper propellor should be placed as close as possible to the feed outlet line so that no slurry build up could gather around the opening. Less maintenance time was needed between runs as catalyst changes could be made by simply pumping the hopper empty and then adding feed and coal to the hopper, mixing them and start running again.

It is noted that the longer coal is left in bitumen, it breaks down and becomes more likely to form a sludge with surrounding particles. It is therefore advisable to add the coal to the bitumen, mix it and pass it through the system as fast as possible having the coal present in the bitumen for as short a time as possible to prevent this. Because of the reasons outlined above and earlier it is reasonable to say that a slurry pump operation permits the evaluation of the disposible coal catalyst concept used for the processing of athabasca bitumen.

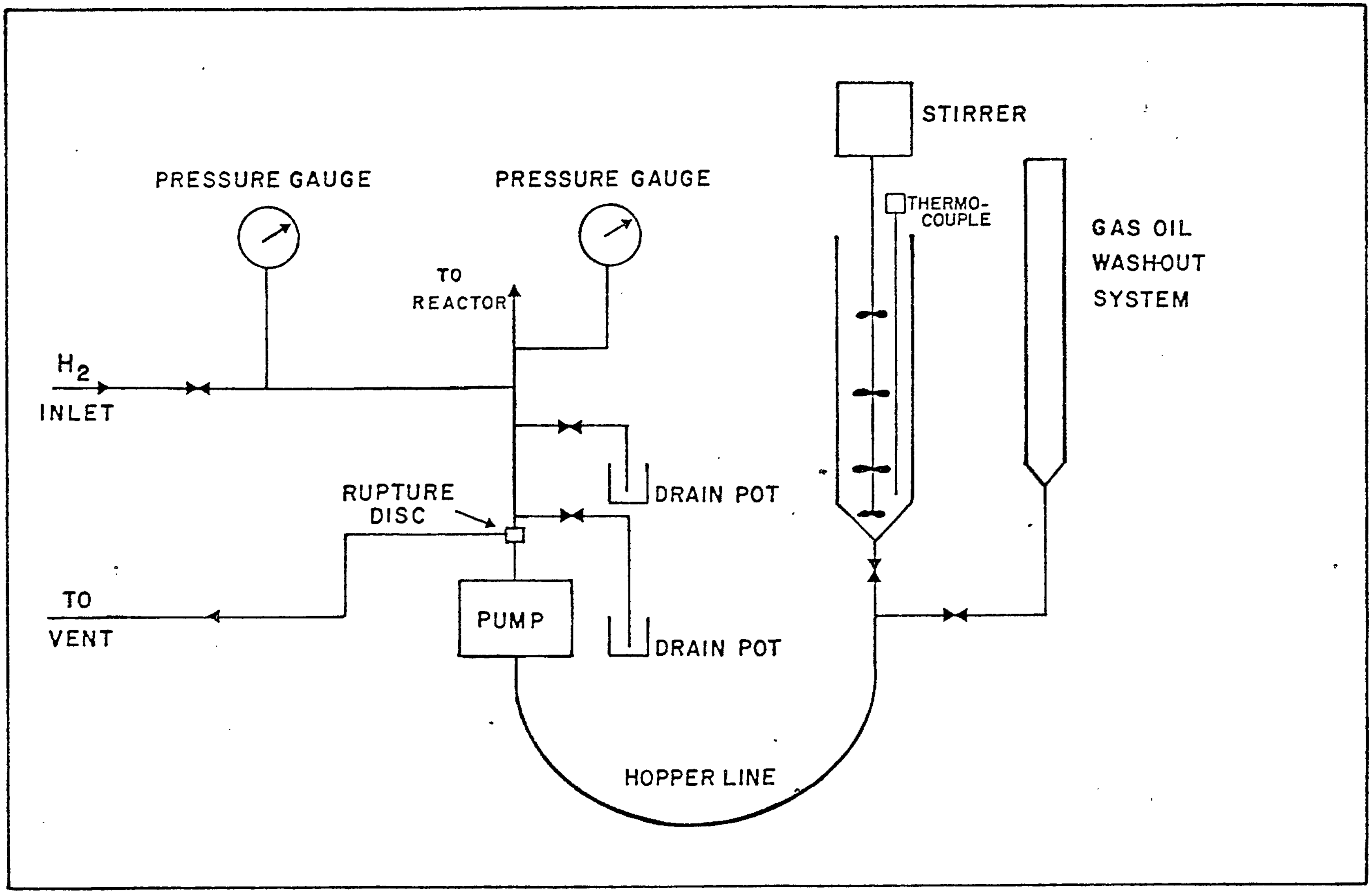

FIGURE - I SLURRY PUMP FEED SYSTEM

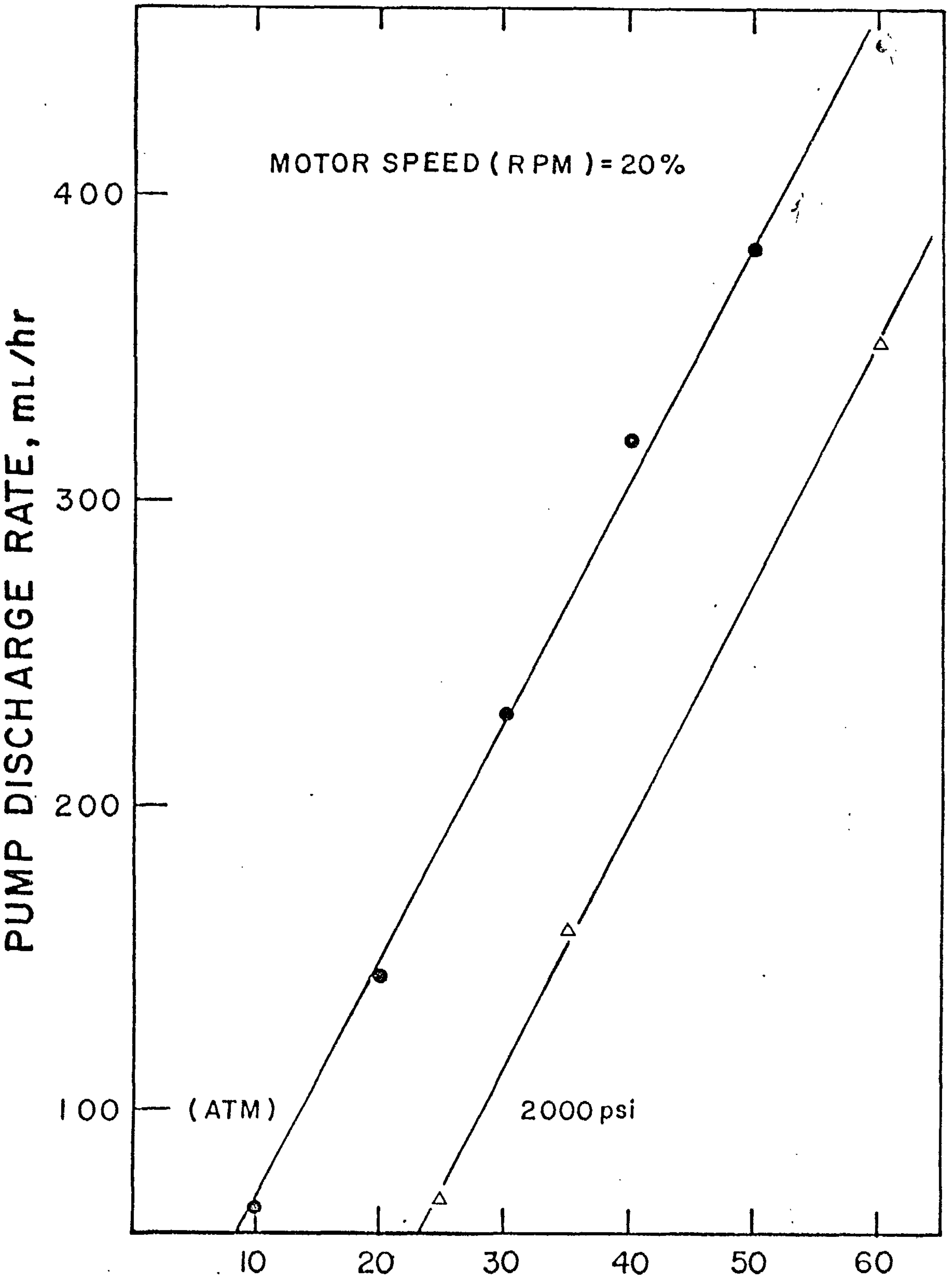

FIGURE-2 PUMP STROKE LENGTH, %

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