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High Moisture Tar at Chicago Plant

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High Moisture Tar at Chicago

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Introduction

Right now, tar quality at Chicago Coke Plant is entirely out of control. Before 1964, there was no tar problem. Since the introduction of Illinois coal in 1965, tar moisture became a problem, and de-emulsifier, RI-3, was used. This method kept tar moisture low for almost ten years. However, tar moisture gradually increased again in 1976. The reason is ^{not clear} still unknown to us; maybe it is due to the deterioration of coke oven ~~and~~ the raise of coking temperature, ^{and on^d off testing of slope charging.} At that time, we found

that the moisture level still could be under control by increasing RI-3 dosage. Since the beginning of this year, RI-3 was found entirely no use at all regardless its dosage. It forced us to work hard to try some other de-emulsifiers. Unfortunately, all de-emulsifiers only worked for a while and only in a specific condition. In the end, we found the basic problem was ^{still} ~~still~~ because of high Q.I. (quinoline insoluble); at high Q.I., de-emulsifier had to be adjusted from time to time in order to fit the

specific coking conditions; such as coke mixture, coking temperature.

Since we could not fool around from de-emulsifier to de-emulsifier, a new general method, slow-stirring method, was developed by our research staff to alleviate this problem. Recently, a sudden sharp jump in solid content raised tar viscosity to such a point that it completely prevented the normal coking operation. A immediate remedy is needed.

A separate supplement PEA are

going to be submitted and it will allow

us to:

1. ~~Find~~ ^{Find} the real cause of high solid tar, and look for ways to solve this problem.

2. Continue on the slow-stirring method.

3. Search for best de-emulsifier after Item No. 1 and No. 2 have been solved, if needed.

Summary

The tar problem at Chicago Coke Plant has been investigated both statistically and chemically. The previous Tretolite de-emulsifier, RI-3, is almost no use at all now. Several other kind of de-emulsifiers has been tried and none of them showed any promising result. Slow stirring proved useful to remove tar moisture, but needed further more work. The search for ^{Cause} ~~Case~~ and prevention on tar insoluble solid is the most urgent work in the future.

Experimental

Tar work we have done so far consists of five main categories:

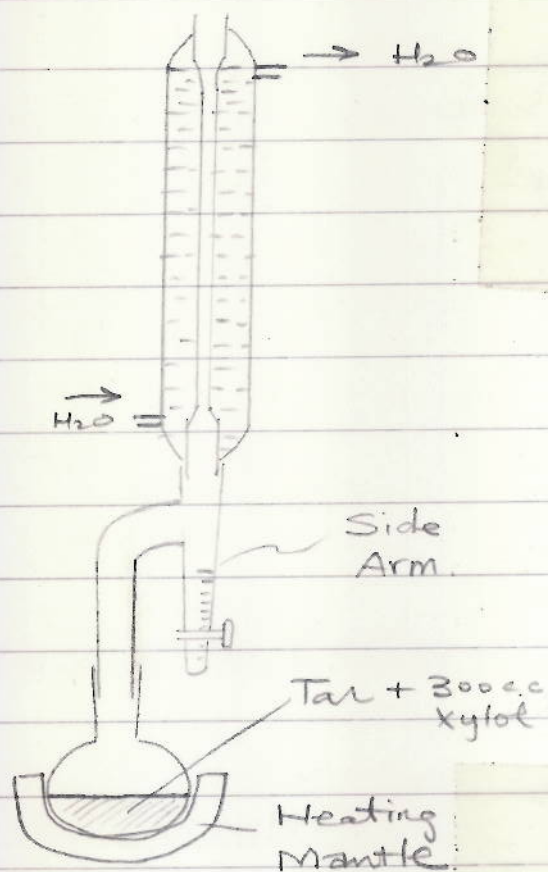
1. Use of different dosage of RI-3 and a trial on Nalco's de-emulsifier 7719 during 1976.
2. Search for new de-emulsifiers during 1977.
3. Development of slow-stirring method.
4. Other de-moisture technique.
5. Search for cause of high insoluble content in tar.

There are two ways to estimate moisture content in tar:

1. Distillation method:

Apparatus is shown in Figure 1.

Fig 1
Distillation Method



Exactly 100 cc. tar and roughly 300 cc. xylol were added into flask. After applying heat, water, as well as xylol, vaporized and condensed in the side arm. Percentage of moisture in tar can be obtained by measuring

the height of water phase in the side arm.

This method is very accurate. However, only

virgin tar was performed this way due to

the limitation of time.

2. Bottle method :

Bottles containing roughly 100 c.c. tar

after adding de-emulsifiers were hand

shaked 60 times. Then, put them in a

water bath maintained around 180°F for 24 hrs.

By measuring the amount of separated water

and comparing with the moisture content in

original virgin tar measured by distillation

method, the moisture content left in treated tar can be obtained. This method is not accurate, and has 3-5% error. However, it is fast, and the amount of error will not hinder our de-emulsifier justification. All our tar de-emulsifiers were being evaluated by bottle method.

The equipment used in "slow stirring" is called "gang" stirrer. It has six stirrers ^{each stirrer} and can ~~stir~~ ^{rotate} at the same speed simultaneously (Figure 2). ^{samples} Tar₁, treated or untreated, were agitated by "gang" stirrers and heated

by water bath at the same time. After 24 hours, measured the amount of separated water and compared with total moisture content from distillation method.

Other de-moisture technique we tried are ① polymers ^{Capable of} forming net-work to catch carbon particles, ②. Ultrasonic vibration to aggregate carbon particles, ③. Ultrafilter to get rid of carbon particles, ④. Pan glomeration to pelletize carbon particles, and ⑤ Various drying reagents. None of them showed any promising result.

Finally, a relationship between Q. I. and amount of tar moisture was correlated through statistics.

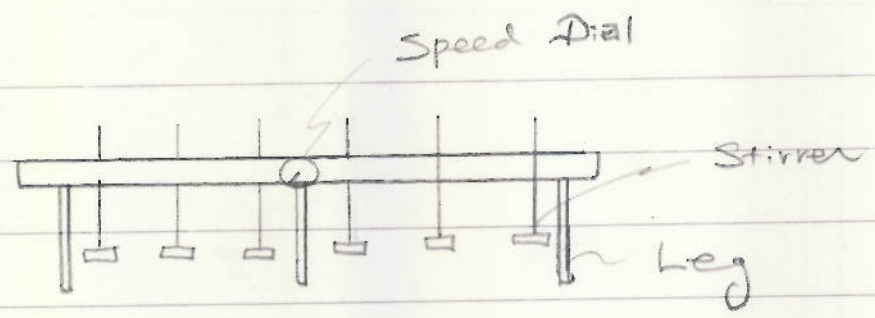


Fig. 2 "Gang" Stirrer.

Future Work

The most urgent job at present is to find out the real cause of increment of insoluble solid content in tar. The

latest laboratory report on ^{high} ash content in mud-like material from gas main suggested that large amount Q.I. is ~~contradictory to that of last year's~~ of fine coal particles may directly or indirectly causes the recent Ash content in Q.I. ~~can~~ ^{may} determine whether serious tar problem.

~~these~~ ^{this} insoluble solid content in tar is

~~directly from coal fine or not.~~ After

that, we should fine out how to avoid

it and restore normal coking operation.

Finally, a continuation ~~re-evaluation~~ ^{method} on slow-stirring ^{and/or}

different de-emulsifiers can be resumed

in order to persuade a high quality ~~to~~

saleable tar.

Results and Discussion

In short, the entire tar work so far is a history of how our research staff faced the increase of insoluble solid in tar. Insoluble solid caused the raise of moisture in tar and also promoted the difficulties to remove tar moisture. Recently, insoluble solid changed tar to such a viscous liquid that normal coking operation became impossible.

Table I shows the increase of Q.I. vs. ^{date} time.

Our tar research project followed the Q.I. trend and changed scope twice. That is why we should discuss it chronologically.

Table I Q.I. Content in Tar

Date	Q.I. Content	Remark
Prior 1964	~2%	Project D2-003-002
1966	~6%	Beginning of using ^{the use of} Illinois Coal
5/5/75 - 12/13/75	8.7%	Composite Truck Tar Sample
12/13/75 - 3/20/76	8.6%	Individual Truck Tar Sample Beginning of test ^{step} charging testing.
5/76	9.0%	Chemical Laboratory Record Composite Truck Tar Sample
6/76	9.6%	" " " "
7/76	10.5%	" " " "
8/76	11.1%	" " " "
9/76	10.3%	" " " "
10/76	12.1%	" " " "
11/76	13.6%	" " " "
12/76	14.2%	" " " "
1/77	16.1%	" " " "
2/77	17.5%	" " " "
3/77	15.0%	Composite Truck Tar Sample. Renew Coal Pulverization facility on 3/15/76
4/77	16.2%	Composite Truck Tar Sample
5/77	20.1%	" " " "
6/77	22.0%	" " " "
6/13/77	35.0%	Production Tar
8/15/77	57%	" "
9/7/77	50%	" "

~~Track tar from Storage Tank.~~

Tar work is usually very difficult, since there are too many variables. Any minor change in coking process or coal mix may change tar chemical composition, and thus the efficiency of moisture removal. That is why tar test seemed never reproducible. All data can only make sense through a statistical point of view.

1. Before 1976

From 1966 to 1975, Q.I. only raised from an average of 6% to an average of 9%. Tretolite's de-emulsifier RI-3

help us to keep tar moisture in line for almost ten years. Starting the summer of 1976, tar took a gradual increase in water content. At that time, after three month testing in site, it was found that heavier dosage of RI-3 was still able to remove tar moisture (Figure 3). Not every production tar sample could be de-watered under this kind of situation. However, by the time tar reached retention tank, they would be well-mixed. 90% trend would be a good approximation for the average tar. That means tar moisture could be maintain

low if 5000 p.p.m. RI-3 or more was used.

At that time, 2000 p.p.m. dosage was the normal dosage. Moreover, tar tested in Figure 3 was heated only 24 hours in water bath. In practice, tar averagely stays hot in decanters for 48 hours and again hot in retention tank for another 24 hours. Therefore, tar moisture would reduce further than what Figure 3 shown. No field test was performed at that time, because winter came and not enough heat could be maintained in decanters.

Nalco's de-emulsifier, 7719, also had

been tested. It worked at exactly 500 ppm.

(Figure 4)

but overdesed at higher dosage₁. There was

no interference between 7719 and RI-3, and

RI-3 could be replaced by 7719 gradually

and smoothly. However, by-product manager

refused to try, because there was no way

he could control the amount of incoming

tar.

Here is a solid evidence that tar

moisture is caused by high Q.I., not through

any personal feeling or by any theory (Figure 5).

Between December 1975 and March 1976,

Chemistry laboratory kept record on moisture

content of every truck tar shipped to Kopper as well as Q.I. on every weekly composite tar. Size of tar tank was the same for every truck, and the composite tar sample was composed from each truck on equal amount bases. During this period, same amount of de-emulsifier, RI-3, was used, so the effect was the same. By averaging the moisture content in each truck tar, weekly tar moisture vs. weekly Q.I. can be plotted. We found that it is very clear that the more Q.I. is in tar, the higher is the left-over tar

moisture. Two more evidences are in Appendix A and B.

2. From November 1976 to June 1977

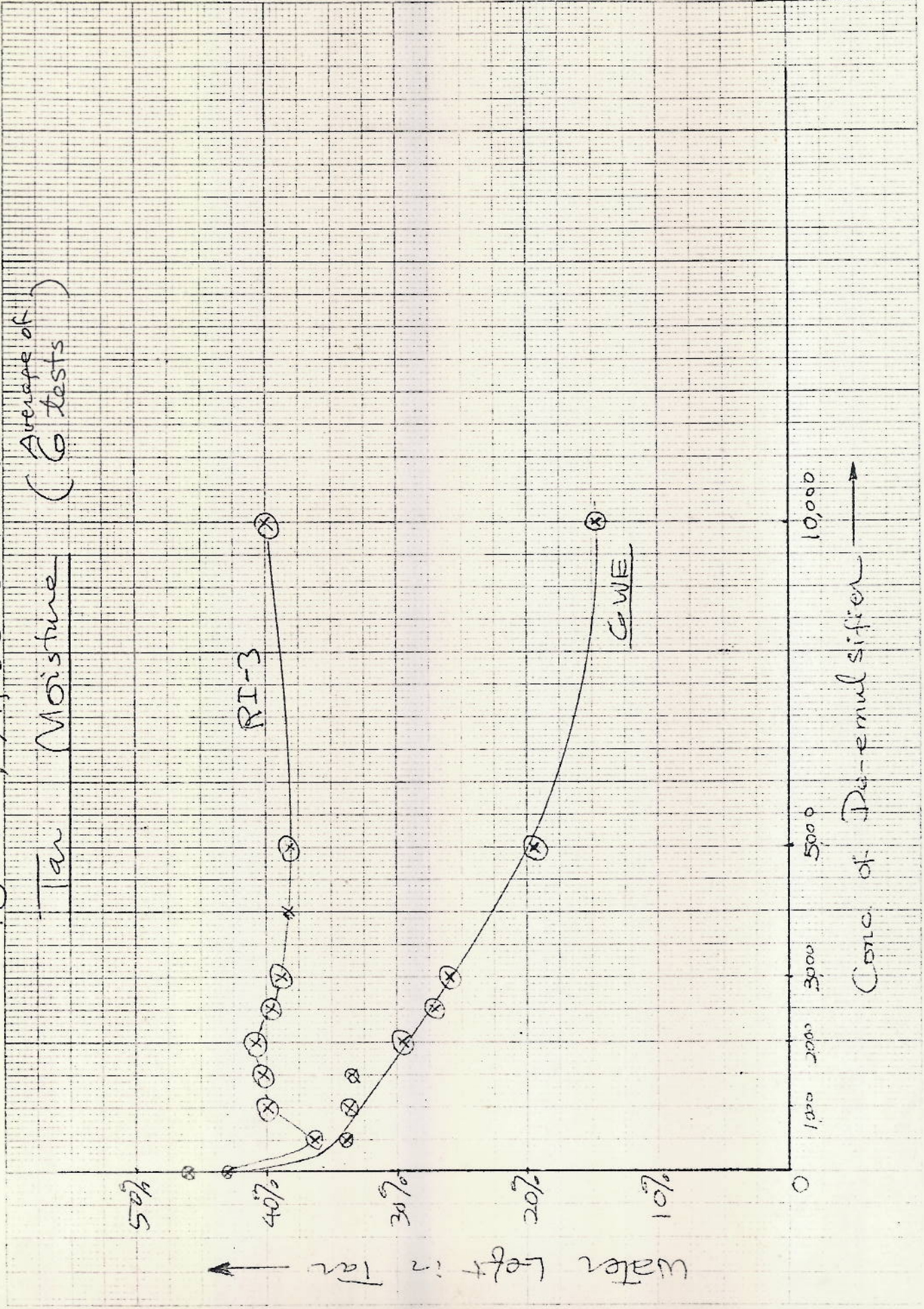
There were two major changes affecting tar quality drastically within this period — stage charging and the reinforcement of coal pulverization. Stage charging became a routine operation, and fine coal particles increased tremendously. Our tar happened to be the victim of these two changes. Q.I. jumped from an average of 12% to 21%. During this stage,

180°F, After 24 hours

Tan Moisture
(Average of 6 tests)

Water Left in Tan

Conc. of Dis-emulsifier



tried — ultrafilter, ultrasonic vibration, pangglomeration, drying reagents, polymer net, -----, None of them was worth to continue. The only one which seemed extremely promising is the slow-stirring method (Figure 8). Slow-stirring can reduce moisture level way below any other methods, with or without de-emulsifier, and always worked well. Why slow-stirring works? The writer does not know much. But, the writer thinks that the agitation may ① promote the aggregation of water drops dispersed within tar, ② Cause Carbon particles

to combine into bigger particles just like
polymer chemist using heat and agitation

to polymerize ^{monomers.} ~~monomers.~~ Slow-stirring

seemed to have a lower limit — moisture
in tar hardly reduced below certain level.

Moreover, slow-stirring was not only a
function of time, but also sensitive to
the shape of paddle. "Whether or not

slow-stirring is a function of Q.I."

and "Which stirring speed is the best"

nobody knows. More research has to be

done before field test. More useful

data are in Appendix E.

3. June 1977 till now.

Further oven deterioration and less oven push raised the Q.I. level in production tar to 35% - 57%*. Tar turned somewhat brown color and became such viscous that it could stop flow any time any place. Tar is no longer saleable. At present, several heaters inserted into decanters and a chemical reagent for reducing tar viscosity was added just to keep ^{tar flow} ~~the coking pro.~~ Gas main filled with mud-like ^{black} material.

A recent laboratory report showed that large
* Q.I. in Production Tar is usually several percentage higher than that of truck tar. It takes almost one month to flow tar from decanter to truck.