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TO: Grover Myers

DATE: January 8, 1989

RE: Patent Disclosure - Catalyst Impregnation in PREMIER

FROM: D. M. Riggs

Over recent weeks, a considerable level of effort has been dedicated towards improving the strength of PREMIER fuels by using a sugar impregnation process followed by a "re-bake" of the fuels. This process is accomplished by first drawing a vacuum on the fuels to remove entrapped air from the fuel pore microstructure followed by impregnation of concentrated sugar solutions into the evacuated pores. The impregnated fuels are then dried and baked at 800°C to convert the sugar in the impregnated fuels to carbon. This process results in a 2 - 2.5X increase in the vertical strength of the fuels. During the course of this work, concern arose over the levels of carbon monoxide (CO) generated by the standard, non-impregnated fuels used in the PREMIER test market products. Given the nature of the vacuum, sugar impregnation process, it was decided that it would be quite easy to impregnated liquid catalysts into the fuel to reduce CO generation at the same time as sugar was being impregnate to increase strength. Several tests to evaluate this idea were run with considerable success. The results of these tests will be discussed herein.

Experimental Procedures

For each test, a total of 100 grams of RJO-13 fuels (lot 300078-A) were placed in a vacuum flask and evacuated for a total for 10 minutes at room temperature to a vacuum pressure of about 25 in Hg. While these fuels were being

evacuated, solutions consisting of 500 ml of water and 150g grams of Dixie Crystals table sugar were prepared. The solutions of sugar and water were kept at room temperature. Depending upon the test, solutions of palladium chloride or platinum chloride were added to the sugar solutions. The platinum chloride was obtained from Fisher Scientific, as was the palladium chloride. The as-received palladium chloride was a 5% solution of palladium chloride in hydrochloric acid. The platinum chloride was a 10% solution of platinum chloride in hydrochloric acid. The test series of impregnations that were run were as follows:

Sample	Catalyst	Amount of Catalyst Added to Sugar Solution	Amount of Sugar Added
1. Control	None	None	None
2. Sugar	None	None	150g/500ml
3. Sugar + Pd	Palladium Chloride	15 ml	150g/500ml
4. Sugar + Pd	Palladium Chloride	50 ml	150g/500ml
5. Sugar + Pt	Platinum Chloride	15 ml	150g/500ml
6. Sugar + Pt	Platinum Chloride	50 ml	150g/500ml

After evacuating the fuels for 10 minutes, the above described solutions were pumped into the evacuated vessel containing the fuels. The solutions were held in the vessel containing the fuels under vacuum for 15 minutes. After this holding period, the vacuum was released and the fuels were placed in the Pulvis fluidized bed for drying. The drying temperature was held at 120°C while the fuels were fluidized with an air flow through the bed of 0.5 cubic meters per minute. This process completely dried the fuels in 10 minutes. The dried fuels were next placed on a 6" wide belt and passed through a Lindberg belt furnace operating at 800°C. The furnace is 15 feet long and the belt was moving at 4 inches per minute. The furnace atmosphere consisted of nitrogen.

After re-baking, some of the fuels were tested for strength and others were used to fabricate hand made PREMIER products. Materials used in the fabrication of the models are shown in Attachment 1. The products were then smoked at standard FTC conditions (35 cc puff every 60 seconds) and their CO deliveries were measured.

Results and Discussion

Shown in the following table are the results of measurements of CO generation and strength measured from the sugar/catalyst impregnated fuels described in the previous section.

Sample	Catalyst	Amount of Catalyst	Amount of Sugar	Vertical Strength Kg	CO Delivered Mg
1. Control	None	None	None	43.9	11.56
2. Sugar	None	None	150g/500ml	93.8	8.41
3. Sugar + Pd	Palladium	15 ml	150g/500ml	86.8	6.30
4. Sugar + Pd	Palladium	50 ml	150g/500ml	-	4.47
5. Sugar + Pt	Platinum	15 ml	150g/500ml	87.6	7.83
6. Sugar + Pt	Platinum	50 ml	150g/500ml	-	5.01

It can be seen from the above table that significant increases in strength and reductions in CO results from the impregnation process. Sample 1, the control, exhibited a strength of 43.9 kg and generated 11.56 mg of CO during smoking. Impregnating with sugar only increased the strength to 93.8 kg and dropped the CO to 8.41 mg with no loss in puff count. Adding palladium chloride solution to the sugar solution at a level of 50 ml per 500 ml of water reduced the CO to 4.47 mg again with no loss in puff count. Similar results can be noted for the platinum chloride solutions. These results are summarized in Figure 1.

The results open a doorway to easily improving two important attributes associated with PREMIER fuels: strength and CO generation. Significant enhancement of strength and reductions in CO can be achieved by using the "dual simultaneous" impregnation process described above. The process itself is quite simple and could be adopted to production in a relatively short period of time.

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