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ENCLOSURE (B) 19

**STUDIES ON PREPARING
FUELS FROM RUBBER**

by

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SUMMARY

Rubber sheet is decomposed by dry distillation to give a high yield of terpene hydrocarbon mixtures as shown below (by volume):

Oil	95%
Residue	1%
Gas and loss	4%

A study was made of rubber decomposition in a continuous cracking pipe-still, and a yield of about 90% of oil and 5% of gasoline was obtained. By hydrocracking the oil product, a 41.6% yield of aviation gasoline, with octane rating of 72.9 clear and 92.4 with 0.15% lead, was obtained.

I. INTRODUCTION

This research was done during the period, November 1943 to June 1944, but the process was not developed commercially because imports of rubber were limited due to transportation difficulties.

II. DETAILED DESCRIPTION

The catalytic cracking pilot plant was used for these tests with the catalyst chamber by-passed. The flow and operating conditions are shown in Figure 1(B)19.

Rubber was cut into blocks about 20x20x5 cm and charged to a direct-fired batch still, where it was heated to about 200°C and melted to fluid which could be pumped. The rubber was in a partially decomposed state which solidified if cooled. The melted rubber was charged to the pipe still and the products were fractionated in the usual way. Experimental results (for Run No.2) are given below:

Process Conditions

Temperature of furnace tube outlet	410°C
Pressure of furnace tube inlet	5 atm
Pressure of furnace tube outlet	2 atm
Total charge of rubber	16,850 kg
Charging rate	240 lit/hr
Recycle ratio	3
Heating time	5.5 min
Heating area of furnace tubes	27 m ²

Yields (% of charge)

Light gasoline	0.2% by wt
Heavy gasoline	35.7%
Kerosene fraction	41.7%
Residual	19.1%
Gas	1.9%
Water	1.3%

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Properties of Blend of Light and Heavy Gasolines

Sp.gr (15/4)	0.822
<u>Distillation</u>	
up to 100°C by Vol	12.5%
100-200°C by Vol	66.7%
200-300°C by Vol	14.7%
above 300°C by Vol	3.1%

Properties of Kerosene

Sp.gr (15/4)	0.936
<u>Distillation</u>	
up to 100°C	0.9%
100-200°C	2.9%
200-300°C	44.4%
300-360°C	42.2%
residue	7.1%

Analysis of Cracked Gas

Hydrogen	16.2%
Methane	34.2%
Ethane	8.3%
Propane	13.0%
Butanes	4.5%
Ethylene	3.0%
Propylene	5.5%
Butylenes	3.7%
C5 or over	4.6%
others	7.3%

The heavy gasoline and kerosene were mixed together and fractionated in the laboratory into the following outs:

Table I(B)19

RESULTS OF FRACTIONATING MIXTURE OF
HEAVY GASOLINE AND KEROSENE

		up to 100°C	100 to 200°C	200 to 300°C	300 to 360°C	Residue	Gas	Water
Yields (wt%)		7.6	26.1	24.0	17.6	15.3	2.5	1.3
Sp.gr (15/4)		0.666	0.817	0.885	0.917	1.001		
Distillation	I.D.	27	86	168	130			
	10%	33	132	203	195			
	50%	37	169	256	319			
	90%	47	194	310	319			
	E.P.	83	241	339	319			
O.V.	plain	80.2	71.2					
	0.15% Pb	81.7	75.7					

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The fraction boiling up to 100°C was hydrogenated. 500cc of charge, 20gm of powdered nickel oxide catalyst, and H₂ under 100 atm. pressure, in a 2400cc autoclave, were heated to 200°C for one hour. The consumption of hydrogen was 178 litres and the properties of the total liquid product were as follows:

Sp. gr (15/4)	0.622
Distillation	
I.D	22°C
10%	28°C
50%	31°C
90%	47°C
E.P	74°C
Octane Value	88
O.V.* with 0.15% Pb	111

*(calculated from 50:50 blend with n-heptane)

The mixed oil of heavy gasoline and kerosene from thermal decomposition of rubber, which had the following properties, was treated in the hydrocracking pilot plant (described in Enclosure (B)16) charging 10 litres per hour.

Sp. gr (15/4)	0.880
Distillation	
10%	140°C
50%	249°C
E.P	395°C

NiO-MoO₃-clay catalyst was used, and the reaction took place at 460°C, 200 atm. and S.V. of 0.5. The yield of reaction product was 103% by volume of charging stock, and the density of the reaction product was 0.796. The reaction product was distilled and the following aviation gasoline was obtained in yield of 41.6% to the charging stock.

Sp. gr (15/4)	0.755
Distillation	
I.D	46°C
10%	78°C
50%	119°C
90%	162°C
97%	171°C
Olefines	4.4%
Aromatics	8.4%
Naphtenes	41.2%
Paraffins	46.0%
Octane value, plain	72.9
Octane value, leaded 0.15%	92.4
Vapour pressure kg/cm ²	0.27

III. CONCLUSION

This gasoline was very clear and sweet, and appeared to be suitable as an aviation gasoline.

Engine tests were not made on this stock, however, and further work on this project was stopped because of limitations in the supply of crude rubber.

No difficulties were noted during the operation of the cracking unit. An examination of the Run 2 showed the furnace tubes to be clean, although some coke was deposited in the bottom of the evaporator and also at the pressure reducing valve. It is believed that the run could have been continued for about 10 days before shutting down due to coking. Difficulties were encountered in the rubber melting apparatus, and Run No. 1 was out short due to

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cooking and smoking resulting from decomposition due to too rapid heating.

To the author's knowledge, cracking of rubber on commercial scale was not done in Japan.

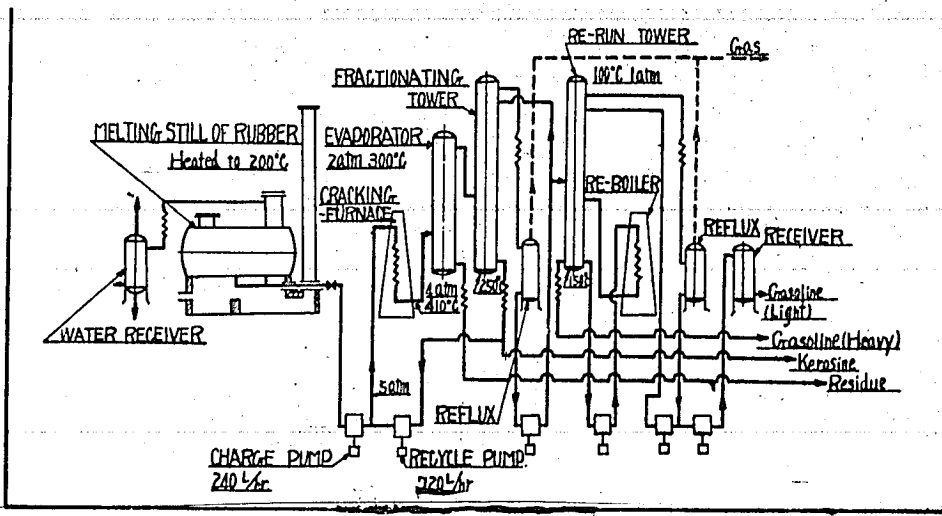


Figure 1(B) 19
APPARATUS FOR CONTINUOUS RUBBER CRACKING