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Studies on the Catalytic Effect of Active Charcoal Impregnated with Nickel

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Summary

Determination of Iodine value of hydrogenated Groundnut oil treated with active sugar charcoal impregnated with Nickel Nitrate solution of different concentrations indicates Nickel in combination with active sugar charcoal acts as an effective catalyst in the process of hydrogenation of vegetable oils, viz. Groundnut oil.

Introduction

The subject "Catalytic Hydrogenation" received its impetus by the discovery of the hydrogen-activating properties of Nickel by Sabatier and Senderens in 1899. Nickel assumed a place of great technical importance in the hydrogenation of vegetable oils, for the last several decades. Extensive experiments on the behaviour of Nickel Catalyst have been carried out by a number of pioneer workers, viz. Benton and White[1], Mori Kawa, Benedict and Taylor[2]; Eucken and Hunsmann[3]; Beeck, Smith and Wheeler[4]; Taylor[5]; Kemball and Taylor[6]; and Brown and Sivasankaran[7].

A survey of the literature reveals that a number of combinations have been applied to use Nickel as a catalyst in the industry, viz.

1. Nickel-Chromia
2. Nickel-Thoria
3. Nickel-Kieselguhr, etc.

But no combination like Nickel-Active Sugar Charcoal has been tried.

[1]) A. F. Benton and T. A. White, J. Amer. chem. Soc. **52**, 2325 (1930).

[2]) K. Morikawa, W. S. Beneditt and H. S. Taylor, J. Amer. chem. Soc. **58**, 1445, 1795 (1936).

[3]) A. Eucken and W. Hunsmann, Z. physik. Chem. **44B**, 163 (1939).

[4]) O. Beeck, A. E. Smith and A. Wheeler, Proc. Roy. Soc. London A **177**, 62 (1940).

[5]) H. S. Taylor, Amer. Scientist. **34**, 553 (1946).

[6]) C. Kemball and H. S. Taylor, J. Amer. chem. Soc. **70**, 345 (1948).

[7]) Brown, C. Herbert and K. Sivasankaran, J. Amer. chem. Soc. **84**, 2828 (1962).

Annexure "A"

Hydrogenation of Groundnut Oil in Presence of

Strength of Nickel Nitrate Solution	Volume of Nickel Nitrate Solution in m. l.	Weight of Active Sugar Charcoal in gms.	Weight of Impregnated and dried charcoal in gms.	Weight of Impregnated Charcoal after Decomposition in gms.	Weight of Impregnated Charcoal after Reduction in gms.	Temperature of Drying of Impregnated Charcoal in °C	Temperature of Decomposition of Nickel Nitrate inside Charcoal in °C	Temperature of reduction of Nickel Oxide inside Charcoal in °C
N	50.000	5.0000	5.0832	5.0126	4.1348	95.100	105—130	300—350
N/5	50.000	5.0000	5.0764	5.0098	4.1156	95.100	105—130	300—350
N/10	50.000	5.0000	5.0938	5.0150	4.1076	95.100	105—130	300—350
N/25	50.000	5.0000	5.0478	5.0080	4.2136	95.100	105—130	300—350
N/50	50.000	5.0000	5.0596	5.0068	4.1518	95.100	105—130	300—350
N/100	50.000	5.0000	5.0576	5.0016	4.0408	95.100	105—130	300—350

N. B.: (i) Groundnut Oil used- Refined, from Swastik Oil Mills Ltd. Bombay (India).
(ii) The experiments have been performed at "Atmospheric Pressure" only.

Annexure "B"

Optical Density and Percent Transmittance

Strength of Nickel Nitrate Solution	N		N/5		N/10	
Experimental condition	Before Impregnation	After Impregnation	Before Impregnation	After Impregnation	Before Impregnation	After Impregnation
Optical Density	0.666	0.630	0.1330	0.1310	0.0710	0.0630
Percent Transmittance	21.58	23.45	73.61	73.96	84.91	86.5

N. B.: Filter used — Red, 600 mμ.

Experimental

Preparation of Active Sugar Charcoal

In a silica basin cane sugar was heated to a high temperature where it melted into a light brown liquid, this on further heating boiled and changed into dark brown liquid. Further at red hot temperature it changed into black elastic semisolid which on continuous heating finally gave black amorphous solid at temperature around 900 °C.

Purification of Active sugar charcoal

Active sugar charcoal was washed with water several times in order to remove any soluble and suspended impurity. The charcoal was then dried at temperature above 100 °C till it was perfectly dry. It was transferred into a desiccator to keep away from moisture.

Nickel-Active Charcoal as Catalyst

Temperature of Hydrogenation of Groundnut Oil in presence of Nickel-Charcoal in °C	Time of Impregnation of Nickel Nitrate Solution in hrs.	Time of Decomposition of Nickel Nitrate inside charcoal in hrs.	Time of reduction of Nickel Oxide inside Charcoal in hrs.	Timeof Hydrogenation of Groundnut oil in presence of Nickel-charcoal in hrs.	Volume of Groundnut Oil used for Hydrogenation in ml.	Iodine No. of Nonhydrogenated Groundnut Oil	Iodine No. of Hydrogenated Groundnut Oil
200—250	24	6	3	3	50.000	94.14	79.51
200—250	24	6	3	3	50.000	94.14	83.47
200—250	24	6	3	3	50.000	94.14	84.55
200—250	24	6	3	3	50.000	94.14	78.87
200—250	24	6	3	3	50.000	94.14	77.73
200—250	24	6	3	3	50.000	94.14	81.90

of Nickel Nitrate Solution

N/25		N/50		N/100		Distilled Water as Reference Solution
Before Impregnation	After Impregnation	Before Impregnation	After Impregnation	Before Impregnation	After Impregnation	
0.024	0.022	0.0130	0.0098	0.006	0.0016	0.0000
94.62	95,06	97.04	97.76	98.62	99.62	100.00

Active sugar charcoal was examined for any possible contamination by performing certain tests for expected anions and cations which were found absent.

Impregnation of Active sugar charcoal with Nickel Nitrate

A definite amount of degassed active sugar charcoal was taken in a well stoppered ERLENMEYER flask, then a definite volume of Nickel Nitrate solution of definite concentration was added. The flask was shaken thoroughly and was allowed to stand for a definite period. During this period the flask was occassionally shaken thoroughly in order to make the impregnation more effective and uniform.

After the recorded impregnation period, active sugar charcoal impregnated with Nickel Nitrate was transferred to funnel fitted with filter paper. The beaker under the funnel

was then replaced by another beaker, and impregnated active sugar charcoal was washed with water till every trace of Nickel Nitrate solution adsorbed was removed from the surface of charcoal.

Drying of active sugar charcoal impregnated with Nickel Nitrate

Active sugar charcoal in combination with Nickel Nitrate was allowed to dry at temperature not more than 100 °C till a constant weight of active sugar charcoal in combination with Nickel Nitrate was obtained.

Decomposition of Nickel Nitrate inside active sugar charcoal

Perfectly dried active sugar charcoal in combination with Nickel Nitrate was heated to a temperature above 105 °C in order to decompose Nickel Nitrate into Nickel Oxide till complete conversion took place.

Reduction of Nickel Oxide inside Active sugar charcoal

Reduction of Nickel Oxide inside active sugar charcoal was brought about by passing Hydrogen, prepared by Zinc and Dil Sulphuric Acid, into distillation flask containing active sugar charcoal in combination with Nickel Oxide at temperature above 300 °C. The process 7 passing of the Hydrogen was continued till almost complete reduction took place.

Hydrogenation of groundnut oil in presence of reduced Nickel inside the Base — Active sugar Charcoal

A definite volume of Groundnut oil was taken into a distillation flask and was heated to near boiling temperature, say 245 °C. Active sugar charcoal in combination with reduced Nickel was then dropped into the oil and was stirred thoroughly. Hydrogen was passed into the oil containing reduced Nickel inside the base — active sugar charcoal. The process of passing of hydrogen was continued for a definite period in order to hydrogenate the oil as much as possible.

Determination of Iodine Number of Oils

Iodine number of hydrogenated as well as non-hydrogenated Groundnut oil was determined by WIJ's method. The details of the method have been taken from "A. I. VOGEL, Elementary Practical Organic Chemistry, Vol. II (Longmans, Green and Co., London) 1958, 767".

Colorimetric Determination of Nickel Nitrate Solutions

Colorimetric determinations of Nickel Nitrate solutions before and after impregnation was made with the help of photo-electric colorimeter. The details of the method have been taken from "A. I. VOGEL, A Text Book of Quantitative Inorganic Analysis, (Longmans. Green and Co., London) 1955, 616".

Observations

See Annexure "A" and Annexure "B".

Conclusion

The observations clearly indicate that the Nickel absorbed (Annexure "B") inside the base-active sugar charcoal acts as an effective catalyst in the hydrogenation of vegetable oils, viz, Groundnut oil, (Annexure "A").

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