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HUA-KUNG HSUEH-PAO

(化工學報)

CHEMICAL INDUSTRY AND ENGINEERING

No. 1, October 1959

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Peking Institute of Petroleum Technology

ABSTRACTS

Platforming of Straight-run Naphthas for
Aromatics and Octane Improvement

This paper deals primarily with the individual platforming of straight-run naphthas (60 - 130° C) from Yümen, Kramayi and Central Szechuen, and hydrogenated shale oil (60 - 120° C) from Fushun for aromatics in an isothermal reactor containing 80 ml of catalyst. Data are given to show the platforming conditions, material balance and product distributions. The aromatic yields from the Yümen, Kramayi, Central Szechuen and Fushun products are 38.5, 31.8, 46.6 and 32.2 per cent in weight, respectively. The yields of the by-products, hydrogen and liquid, are 1.7 - 2.2 and 90 - 94 per cent. In the course of a 2,170-hour lifespan test in an activator containing 50 ml of catalyst, the volume of the aromatic yield decreases from 38.8 to 35.4 per cent.

The paper also presents a preliminary study of the improvement of the octane rating of straight-run Yümen and Kramayi gasolines (80 - 180° C). At a yield of 90 per cent in weight, the motor-method octane number of the debutanized product increases from 44 (feed stock) to 72.

The catalysts used in these experimental processes were developed at the Research Institute of Petroleum Science, Ministry of Petroleum Industry.

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High Purity Toluene Recovery from Platformate
by Extractive Distillation with Phenol

This paper presents a pilot plant study of high purity toluene recovery from platformate by extractive distillation with phenol. The extractive distillation column used in the study is 200 mm in diameter and is equipped with 44 bubble-cap plates. It has a capacity of processing 250 kilograms of feed per day. The platformate is first prefractionated to 70 - 119° C, containing in weight 0.8 per cent benzene and 48 per cent toluene. The heart cut is then used as the feed for the extractive distillation. The recommended operating conditions are: phenol/feed ratio, 4; reflux ratio, 4 - 5; feed temperature, 120° C; phenol inlet temperature, 130° C. The actual number of plates in the rectifying, stripping and phenol recovery section of the column are 19, 20 and 5, respectively. The recovery of nitration grade toluene from the feed is 97 - 98 per cent. When losses in prefractionation, acid washing and redistillation are included, the net recovery of toluene from the platformate is 93 per cent.

The result of this study affords ample data for the design of full scale production plants.

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Simplified Method for Refining Low Temperature Coal Tar
by Extraction with Sodium Phenolate Solution

This paper presents the study of a method of simplifying the process of refining low temperature coal tar fractions and lowering the consumption of acid and alkali by extraction with sodium phenolate solution.

In the course of the experimentation, investigations are conducted on the influences of the NaOH solution concentrations and of the phenol contents in the coal tar fractions on (1) the effectiveness of the extraction, (2) the quantity of NaOH required during countercurrent extraction, (3) the quantity of water required to dilute the saturated phenolate solutions obtained for the separation of the dissolved phenols, and (4) the physico-chemical properties of the refined oils and crude phenols.

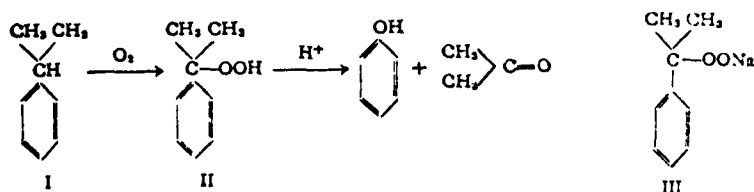
The experimental results show that in the phenolate extraction process, only 10 - 15 kg of NaOH is required to extract 100 kg of phenols from the coal tar fractions. The quantity of NaOH used here is much less than that used in the ordinary alkali extraction process, in which 40 - 45 kg of NaOH is required to extract 100 kg of phenols. The refined oil can be used as diesel fuel without further refining while the phenols of both higher and lower boiling point can be separated by diluting the extracts. It was also observed that an accompanying effect of lowering the sulfur and nitrogen contents of the fractions appears significant.

The results of a number of tests of the refined oil made in diesel engines of 1200 and 2500 rpm show that the quality of the oil is satisfactory.

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Auto-oxidation of Cumene

A preliminary study on the effect of the addition of cumene hydroperoxide (II) with or without the simultaneous addition of an alkaline additive (sodium carbonate or calcium oxide) on the reduction of the induction period in the auto-oxidation of cumene (I) showed that the heterogeneity inherent in these systems resulted in poor reproducibility. Sodium salt of cumene hydroperoxide (III) was introduced as an oil-soluble system containing a combination of the initiator and the alkaline additive. Experiments with various amounts of III at 110°, 120°, and 130° showed that the induction period and the auto-catalysis are either completely or practically eliminated. The reproducibility of the experiments accompanying the use of III was excellent. The rate of accumulation of II was $16 \pm 0.7\%$ (weight)/hr at 130°, and 8.7%/hr at 120° except that in the latter case when the amount of III was 3.33%, the rate decreased to 7.3%/hr. At 110°, the rate varied more pronouncedly with the amount of III, i.e., 3.0 - 5.0%/hr.



Oxygen-absorption in a closed system was observed in order to examine more closely the phenomena of the elimination of the induction period. It was found that a rapid absorption of oxygen took place in the first minutes, followed by a somewhat slower absorption. Kharasch's conceptions on the alkaline catalyzed decomposition of II and the

subsequent reactions between the resulting dimethylphenylcarbinol and II, yielding free radicals, were considered to be adequate in explaining the instantaneous initiation of the reaction chain. Water of crystallization in II facilitates the ionic reaction. Addition of solid sodium hydroxide also caused a rapid initial oxygen absorption, but a very brief induction period was observed. This is believed to follow the same course of reaction as outlined above, with traces of II participating in I.

The efficiency of the auto-oxidation of I with respect to the accumulation of II when III was used was found to be 93 - 97%.

The use of cobalt stearate in promoting the rate of auto-oxidation of I was investigated. At 110°, addition of the cobalt-salt indeed increased the initial rate of accumulation of II (11.5 - 12.5%/hr), but the same mechanism that induced the reaction chain and subsequently brought about the faster rate of reaction prevented the accumulation of II (25%) to a higher degree. The destruction of II by cobalt ions was evidenced by the fact that, with the highest concentration of cobalt-salt used, there was actually no accumulation of II at the beginning of the reaction. Addition of II together with cobalt-salt did not further promote the reaction. It is concluded that traces of II in I were sufficient to be responsible for the chain-initiation processes.

The lower accumulation of II with the use of cobalt-salt was remedied by simultaneously adding III. The result was that the rate of accumulation of II at 110° corresponded to that at 130° with the addition of either II or III. The degree of accumulation of II was also high (40%).

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HUA-KUNG HSÜEH-PAO

(化工學報)

CHEMICAL INDUSTRY AND ENGINEERING

No. 2, December 1959

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Chu Tze-she (朱子系)

ABSTRACTS

Pilot Plant Studies of Platforming in
Multiple Adiabatic Reactors

In order to obtain process design data for platforming plants, pilot plant studies were carried out in a multiple adiabatic reactor system containing a series of three or four reactors. The pilot unit has a daily charge capacity of 200 to 350 kilograms of naphtha. The charge stock was 85 - 125° C cut of straight-run Yümen gasolines for toluene production. A heat balance method based on the compositions of the charge stock and platformed products was developed to evaluate the temperature drop in each reactor. The results indicated that each reactor reached adiabatic conditions. The three-reactor system operation gave a 25 per cent toluene yield (weight based on feed), corresponding to 82 per cent of the possible maximum from the conversion of naphthenes in the charge. Liquid yield of the platformate was 92 per cent (weight), and the byproduct hydrogen had a purity of 95 per cent (volume).

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Kinetic Studies on the Oxidation of Sulfur Dioxide
on Vanadium Catalyst

III. Comparison of Rate Equations Obtained
with Industrial Vanadium Catalysts

Rate equations obtained with a number of industrial vanadium catalysts for the oxidation of sulfur dioxide showed complexity of the kinetic reaction. Although the theoretical rate equation for this type of catalytic process remains uncertain, among the empirical equations, the following is preferred for design purposes:

$$\frac{dC_{SO_2}}{d\tau} = k_1 C_{O_2}^{1/2} C_{SO_2}^{1/2} - k_{-1} \frac{C_{SO_2}^2}{C_{O_2}^{1/2} C_{SO_2}^{1/2}}$$

or

$$\frac{dC_{SO_2}}{d\tau} = k C_{O_2}^{1/2} (C_{SO_2} - C'_{SO_2})^{1/2}$$

IV. Internal Surface Available
in the Industrial Vanadium Contact Mass

The fraction of the internal surface available for industrial vanadium catalysts in the oxidation of sulfur dioxide is rather small and becomes even smaller at higher conversions. Data from both laboratory experiments and industrial practice supported this view. The new fact presented by the author of this paper on the surface available in the catalyst indicates that hollow-catalyst can be used in any part of the industrial converter and gives much higher efficiency.

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Vulcanizing of Thick Rubber Goods

Methods are devised for calculating the change of the inner temperature gradient of rubber goods during vulcanisation regardless of whether or not the temperatures at both sides of the conducting medium correspond.

Although the problem concerning the change of the inner temperature gradient of complex rubber goods, such as automobile tires, during vulcanization has already been solved by foreign rubber technologists, the calculation of the gradual increase of temperature in the vulcanization of tires, that is, the change of the external heat with respect to time, has not been successful up to the present. It is now found that the results calculated by applying the heat-conducting equation for rubber sheets with introduction of the concept of "synthetic" or "equivalent" numbers for complex rubber goods are in close approximation to the actual determination.

Three charts are provided in order to simplify the calculation. Methods of calculating the vulcanizing effect and devising vulcanizing techniques are also suggested.

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Boiling Heat Transfer Coefficients of
Binary Liquid Mixtures

The single horizontal tube evaporator used in this study which was described in a previous article by Lin Chi-fang (林紀方), Yang Yu-ch'i (楊友猷) and K'ung Fan-kuo (孔繁果), Journal of the Dairen Institute of Technology No. 1, 1959, and Chemical Industry and Engineering, No. 2, 1958, was modified by a vacuum system in order to operate the boiling experiments under diminished pressures.

The binary liquid mixtures employed are water-ethanol and benzene-toluene. The conditions of the experiments are as follows:

- (1) Heat load: $q = 4,000 - 40,000$ kcal/(m²) (hr) (C°);
- (2) Operating pressure: $p = 200 - 760$ mm Hg;
- (3) Compositions of binary liquid mixtures:
 - (a) Percentage of benzene in benzene-toluene system:
0%, 12%, 25%, 50%, 75%, 88%, 100%;
 - (b) Percentage of ethanol in water-ethanol system:
5%, 25%, 60%, 91.8%.

The boiling heat transfer coefficients of water under various pressures were examined as preliminary experiments, the results of which may be correlated as follows:

$$\alpha = 4.0 p^{0.2} q^{0.69}$$

For pure benzene, we obtained the empirical formula,

$$\alpha = 4.5 p^{0.2} q^{0.6},$$

and for pure toluene,

$$\alpha = 3.1 p^{0.17} q^{0.6}$$

The heat transfer coefficients of the boiling mixtures of different compositions and under different pressures were correlated with the dimensionless groups suggested by G. N. Kruzhilin. We propose that his original equation,

$$N_s = 0.082 P_r^{-0.45} K_p^{0.7} K_s^{1/3},$$

be used for binary liquid mixtures in the modified form,

$$N_s = 0.71 P_r^{-0.45} K_p^{0.7} K_s^{1/3},$$

with less than $\pm 10\%$ error.

We also correlated our experimental results with S. S. Kutateladze's equation,

$$N_s = 10.9 \times 10^{-4} P_r^{0.5} Re_s^{0.7} K_p^{0.65}.$$

For binary liquid mixtures, we propose the modified form,

$$N_s = 47 \times 10^{-4} P_r^{0.5} Re_s^{0.7} K_p^{0.65},$$

with around $\pm 10\%$ error.

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Boiling Heat Transfer Coefficients of Liquid Metals

A single tube evaporator was designed for studying the heat transfer coefficients of liquid metals at either horizontal or vertical position. The heating element, the evaporating vessel and the condenser were made of stainless steel.

The liquid metals employed were mercury and amalgams containing different quantities of magnesium and sodium. The boiling heat transfer coefficients were determined under pressures from 1 - 11 atmospheres (abs.), heat load from 5,000 - 47,000 kcal/(m²) (hr), and boiling temperatures from 356 - 524^o C.

For mercury, we obtained the following relations:

$$1 \text{ atm. (abs.):} \quad \alpha = 4850 \cdot q^{-0.26};$$

$$4 - 11 \text{ atm.:} \quad \alpha = A \cdot q^{0.46}.$$

Similar results were obtained for amalgams.

The experiments were carried out under N₂ atmospheres.

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(化工學報)

CHEMICAL INDUSTRY AND ENGINEERING

No. 2, June 1960

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Behavior of the Viscosities Prepared from Different Cellulosic Materials
during the Ripening Process

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Fundamental Properties of the Flow of Granular Materials through
Apertures

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Vapor-phase Hydrogenation of Furfural to Furfuryl Alcohol in a
Fluidized Bed

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Absorption Action of Sulfonated Coal for Phenol Homologues

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Tientsin Institute of Technology

Separation of Anthracene, Phenanthrene and Carbazole from Crude

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Hua-tung Chemical Engineering College

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 Chien Hsüan-chih (簡 璣 芝)
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Research Notes

Polymerization of Nylon 66 at Ordinary Temperature and Pressure

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Preparation of Ammonium Fluoride by Neutralization of Fluosilicic

Acid with Ammonia

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ABSTRACTS

Behavior of the Viscoscs Prepared from
Different Cellulosic Materials during
the Ripening Process

The chemical and colloidal changes and the distribution of xanthate groups of the viscoscs prepared from three different cellulosic materials, namely Finnish sulfite wood pulp, prehydrolyzed sulfate bagasse pulp, and prehydrolyzed soda cotton linters pulp, were studied during the ripening process. The results indicate that different sources of cellulosic materials have almost no influence upon the rate of change of the γ value (degree of xanthation) or the electrolyte value (coagulation property). The relations between the γ value or the electrolyte value (E) and the time both fit into the equation of the second-order reaction. At 20° C, the velocity constants for the three viscoscs are approximately the same, i.e., $K_{\gamma} = 1.87 \times 10^{-4} \text{ (hr)}^{-1}$ and $K_E = 7.95 \times 10^{-5} \text{ (hr)}^{-1}$.

The relation between the electrolyte value and the γ value of the three viscoscs shows that at the same electrolyte value, viscose from the wood pulp has the highest γ value, followed by that from the bagasse pulp and that from the cotton linters pulp in a decreasing order. This relation is connected with the distribution of xanthate groups of the viscoscs.

The determination of the distribution of xanthate groups according to the Scherer-Phillips method shows that the viscose from the wood

pulp has the most uniform distribution, followed by those from the bagasse and the cotton linters pulps in a decreasing order. The fractionation of the diethylchloroacetamide derivative of cellulose xanthate using the chlorohydrin-water system depends not only on the γ value, but also probably on the degree of polymerization.

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Fundamental Properties of the Flow of Granular Materials through Apertures

The flow of granular solids through apertures was studied under the following three conditions:

- (1) With no pressure differential,
- (2) under positive pressure differential, and
- (3) under negative pressure differential.

The influence of various related factors upon the solid flow rate was determined. Based upon the correlation of 665 sets of data, the following equations were obtained:

For calculating a flow rate with no pressure differential:

$$G_0 = C_1(\rho_s d_p^{2.5}) \left(\frac{d_0}{d_p}\right)^{2.4} \mu^{-0.5}$$

where $C_1 = 0.66$ when G_0 is expressed in kg/sec.

For calculating a flow rate under positive pressure differential:

$$G_1 - G_0 = C_2(\rho_s^{1.25} d_p) \left(\frac{d_0}{d_p}\right)^{1.4} \left(\frac{\Delta p}{\rho_s d_p}\right)^{0.43} \left(\frac{d_p}{H}\right)^{0.5}$$

where $C_2 = 2.3 \times 10^{-4}$ when $G_1 - G_0$ is expressed in kg/sec.

For calculating a flow rate under negative pressure differential (before the critical point):

$$\frac{G_0}{G_c} = C_s \bar{v}_c / L$$

where $C_s = 5.04$ for sand and 9.84 for coal.

For calculating the sealing pressure differential gradient of a vertical stand-pipe:

$$\frac{\Delta p_k}{L} = 7.35 \times 10^{-2} \rho_p - 10 \text{ mm Hg/m.}$$

.....

Vapor-phase Hydrogenation of Furfural to Furfuryl Alcohol in a Fluidized Bed

Investigations were made on the catalytic hydrogenation of furfural to furfuryl alcohol in a fluidized bed. Effects on the reaction of the variables, such as temperature, inlet gas composition and space velocity, were studied. The optimum reaction conditions shown by the experimental results are:

Temperature: 140°C ;

Space velocity: 2.6 - 3.0 liters/min/100 ml catalyst;

Inlet gas composition (molar ratio of furfural to hydrogen):

1 : 40 - 45.

The once-through yield of furfuryl alcohol is greater than 90%, and the outlet mixture contains no unreacted furfural.

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Absorption Action of Sulfonated Coal
for Phenol Homologues

Sulfonated coal possesses an excellent absorption action for phenol and its homologues. In the present experiment the absorption rates, the equilibrium concentrations, and the factors affecting the absorption action of sulfonated coal for phenolic compounds are systematically investigated. The results obtained show that the absorption rates of sulfonated coal for phenolic compounds are very rapid and that equilibrium is reached after 90 minutes. Among the homologues, the absorption of phenol is preferential. The experiment proves that absorption is, substantially, the action of the active center of sulfonated coal upon the hydroxyl group in the aromatic rings. Applying the above conclusions, the approximate total quantity of phenolic compounds absorbed by sulfonated coal may be obtained when various samples of waste water of different phenolic compound content are treated with sulfonated coal. When the operation temperature is raised to 70° C, the absorption concentration noticeably decreases, but low temperatures (< 10° C) are also disadvantageous. Generally, a temperature in the range of 20 to 30° C is favorable. It is obvious that high temperatures favor the desorption of phenolic compounds from sulfonated coal. When the pH value of the solution exceeds 8, the absorption of phenolic compounds by sulfonated coal drops sharply. The presence of sulfides in solution weakens the absorption of phenolic compounds by sulfonated coal due to the formation of phenolates. Other impurities such as amino- and cyano-compounds as well as organic acids have no apparent influence if their concentration

is not higher than their usual content in waste water. Excessive alkali in solution apparently suppresses the absorption ability of sulfonated coal for phenolic compounds, and even blocks the action entirely. In short, either in view of the absorption rate or of the factors affecting the absorption process, the facts obtained prove that the dephenolizing action of sulfonated coal is an absorption rather than an ion-exchange process.

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Vapor-liquid Equilibrium of the Ternary System,
Toluene-n-Heptane-Heptene-(3)

The vapor pressure of heptene-(3) was determined. The vapor-liquid equilibrium of two binary systems, toluene-heptene-(3) and n-heptane-heptene-(3), and of the ternary system, toluene-n-heptane-heptene-(3), was also determined by using the Gillespie type equilibrium still. With the equilibrium data of three binary systems, n-heptane-toluene, n-heptane-heptene-(3) and toluene-heptene-(3), the vapor-liquid equilibrium of the ternary system, toluene-n-heptane-heptene-(3), was calculated by means of the Spinner and White equations, respectively. The calculated results were found to coincide with the experimental data.

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