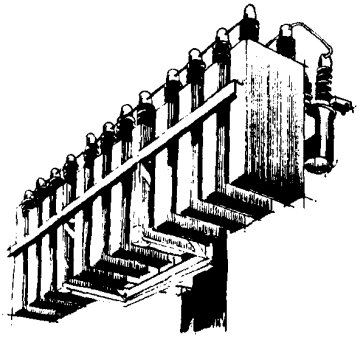
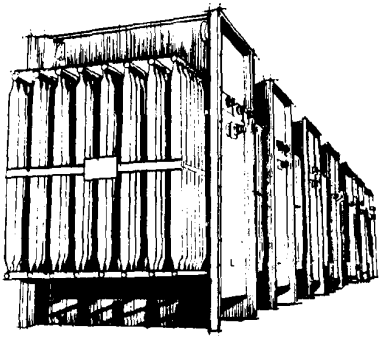
Adsorptive Refining Of Askarel Fluids



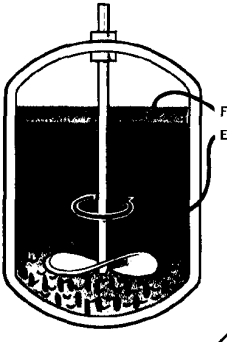


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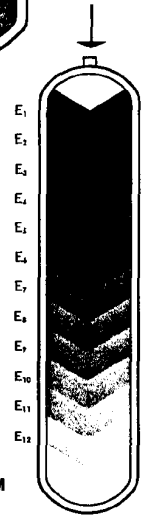
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Adsorptive Refining Of Askarel Fluids



BATCH SYSTEM
Figure 1



COLUMN SYSTEM
Figure 2

Introduction

The performance of transformers and capacitors is generally based on the power factor. This quality is dependent upon the removal of adsorbable impurities.

For many years Monsanto Company factories, as well as many customers have refined askarel fluids by the simple agitation of a given quantity of Fuller's Earth with the fluid to remove adsorbable matter. The treatment was followed by filtration to separate the adsorbent from the refined fluid. When an adsorbent and fluid containing adsorbable matter reach equilibrium, the equilibrium generally conforms to the Freundlich isotherm

$$\frac{x}{y} = kC^m$$

where:

x = the quantity of adsorbate,

y = the quantity of adsorbent used,

C = the concentration of the adsorbate remaining in the fluid at equilibrium, and

k, m = constants characteristic for a specific system.

The isotherm indicates that on one single equilibrium contact, a high degree of purification cannot be achieved. The equation indicates further that with a sufficient number of repetitive contacts, the value of C may be made to approach zero (absolute freedom from adsorbable material). Since it is not practical nor possible to carry out sufficient repetitive batch refinements to achieve the maximum quality, one must resort to the next most practical approach; that being a packed bed which achieves these repetitive treatments on a single pass. To accomplish this, it is necessary to select the proper adsorbent and design a system with enough zones, which we shall refer to as equivalent transfer units (ETU), for maximum purification. The sketches show a visual comparison between the batch and the packed bed system:

As shown in Figure 1, the batch system reaches only one equilibrium, that being dependent on the amount of adsorbable impurities in the fluid and the capacity of the adsorbent. The column system, Figure 2, shows the numerous equilibriums reached as the fluid passes through the column. When material passes through successive zones, the adsorbate collects in the bed. In each zone the consistently cleaner askarel contacts fresh adsorbent. In this way maximum purity is obtained.

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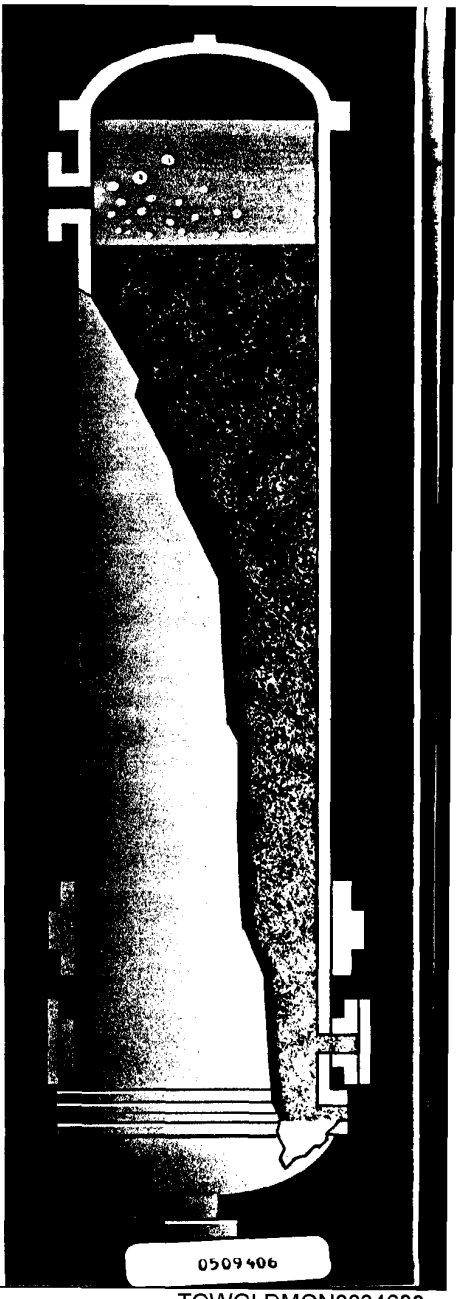
Adsorbent Selection and Activation

Although Fuller's Earth (Attapulgus clay) has been used for years as the selected adsorbent, it does not function satisfactorily in the column system because of its softness. The granular material crushes and forms a mud when wetted with the fluid. The mud formation in a packed bed system restricts the flow to the point where the system is inoperative.

To overcome the problems presented by the attapulgus clay and to achieve other desired features, Porocel*, after extensive testing, was selected as the proper adsorbent. Porocel, being an activated bauxite, is considerably harder than the attapulgus clay which prevents crushing and even the fine material doesn't form a mud. Additionally, Porocel has about 36% more surface area for effective adsorption than does the clay. Best performance was achieved with 20/60 mesh although 30/60 or even 60/90 mesh material will probably give satisfactory performance. The −60 or 200+ mesh is definitely not recommended. At the other extreme, coarser than 20 mesh is not recommended.

Before any adsorbent can be used for refinement, it must be thoroughly activated. The "degree of activation" is best determined as water content. For Porocel an acceptable moisture level is less than one percent (via Karl Fisher determination). If the adsorbent is packaged and shipped under fairly moisture tight conditions such as polylined bags or steel drums, very little drying is necessary. The adsorbent can be dried in one of several ways. First, the adsorbent can be heated in shallow drying pans or a rotary kiln to a temperature of 450°F to about 900°F at atmospheric pressure and then charged to the adsorber. Second, the adsorber can be charged, vacuum applied (5 millimeters of mercury) and the adsorber temperature driven to about 300°F. A third method, and probably the most expensive, is to fill the adsorber, heat to about 300°F, and purge with dry N_2 or dry air until the moisture is driven off. Since most plants handling askarel fluids have steam and vacuum facilities, the combination of heat and vacuum is probably the most economical way to dry the adsorbent. After the drying operation, the heat must always be cut back depending on the askarel being refined and the recommended temperature for refinement.

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Adsorption Theory

Each adsorbent granule, after activation, consists of a myriad of submiscroscopic pores running throughout the granule. Along the walls of these pores lies the active adsorptive surface. As an example of the great surface area and the smallness of the pores, one pound of Porocel contains more than 24 acres of surface. The same equivalent for Fuller's Earth is 13 acres. The fitness of an adsorbent for a specific application is determined by the pore size, pore size distribution, pore volume, and surface energy.

The volume of a particle is made up of the volume of the pores (I_p) plus the volume of the solid forming the balance of the granule. The density of the solid material excluding the pore volume is known as the crystalline density (D_c). Including both solid and pore volume is called particle density (P_d). The density of a particle is the reciprocal of its volume.

In any column system you are concerned with a large mass of individual particles of irregular shape. The particles usually vary in size and size distribution between specified mesh limits. Due to the irregular shape, it is extremely difficult to obtain a three dimensional measurement of these particles. Therefore, most mathematical explanations of packed bed columns are based on a shape factor (Q_s) and a mean equivalent spherical diameter (D_p). This concept has allowed mathematical forecasting of many results with engineering accuracy. It is the equivalent to treating the mass as if it were composed only of spherical particles of an average size. The mean equivalent spherical diameter is usually computed from the screen analysis of the adsorbent.

In a packed bed, all the space between the granules is not filled. These intergranular spaces are called voids (E) and must be differentiated from the pores within the individual granules. It is through the voids that the flow passes and it is through the pores that the mass achieves its adsorptive capacity. The bed density (B_D) includes the space occupied by the voids and that occupied by the mass of the particles.

By applying vibrational energy during the packing of a fixed bed, about 15% more adsorbent may be placed in the same volume than by simply dumping the adsorbent. This can result only from a decrease in the void space with the consequent increase in the bed's effective density (B_D). Such compaction of the bed must affect the course of adsorption since the void space forms the channels through which the fluid flows. The closer packing of the granules tends to reduce the transfer surface at the solid-fluid interface, and at the same time, increases the uniformity of the void channels. For mathematically defining the flow, the average size of the void canals is assumed to be equal to the mean effective particle diameter.

When the fluid containing adsorbable constituents is passed through the bed, the flow is distributed in many different directions from that which it is introduced. Therefore, a given portion of the fluid may travel an extremely tortuous path and be subjected to continuous remixing with other portions of the fluid. The relatively rate of flow of such a portion of the fluid as compared to any other portion of the fluid is determined by the uniformity of the void canals. The canals are most uniform when the bed is packed to its optimum density.

Adsorption is a diffusion mechanism which does not occur instantly. The adsorbate must diffuse first through the body of the fluid to the interfacial film between the solid adsorbent and the fluid. It must then diffuse through the interfacial film and finally by capillary diffusion reach the active adsorptive surfaces on the interior pores. Of the three, the capillary diffusion is the slowest and, hence, controls the rate of adsorption. Since the distance to be traveled by capillary diffusion is determined by the dimensions of the adsorbent particle, final rates must consider the particle size and the exterior area of the particle available for mass transfer. With respect to the fluid, the rate of diffusion (D_L) is governed by adhesion tension, interfacial tension, and the viscosity of the fluid (μ). Only the viscosity can be evaluated accurately, therefore the quantitative treatment of adsorptive rates must be somewhat empirical.

We have considered only what affects the rate of adsorption. The capacity of the adsorbent bed is dependent upon the pore size distribution and pore volume. The degree of purification is a function of the number of equilibrium contacts (ETU), i.e., the height of the bed (H). With these factors in mind we are now prepared to consider some practical evaluation of these principles.

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Operational Temperature

As stated in adsorption theory, "invariably an increase in temperature leads to a decrease in the amount adsorbed, and vice versa",[1] the temperature becomes an important operating variable. From actual experience, the optimum temperature for maximum refinement has been found to vary depending on the fluid. Usually the operating temperature is a function of the lowest temperature at which a reasonable fluid viscosity (μ) can be maintained. As one may expect, because of poor diffusion (D_L), if the fluid is too viscous, the degree of refinement will actually drop. Therefore, temperature control in adsorptive refinement becomes mandatory. From actual experience we have found the following temperatures to be most suitable for refining the specific fluid.

Fluid	Temperature (°C)
Aroclor 1260	135
Aroclor 1254	116
Aroclor 1248	85
Aroclor 1242	70
Mix of Aroclor 1260	30
TTCB, TCB, and dicyclodiepoxy carboxylate	
Trichlorobenzene	25

(1) "Fundamental Principles of Physical Chemistry," Pratton and Maron.

DESIGN CALCULATIONS

Nomenclature for Symbols Used in Calculations

B_D =	Actual bulk density based on the bed	lbs/ft³
E =	Voids Fraction	dimensionless
P_d =	Particle Density	lbs/ft³
D_c =	Crystalline Density	lbs/ft³
I_p =	Internal Porosity	%
D_B =	Bulk Density	lbs/ft³
HETU =	Height Equivalent Transfer Unit	ft
ETU =	Equivalent Transfer Unit	dimensionless
J_D =	Chilton — Colburn j — factor for mass transfer	dimensionless
J_H =	Chilton — Colburn j — factor for heat transfer	dimensionless
R_e =	Reynolds Number	dimensionless
S_c =	Schmidt Number	dimensionless
k_x =	Mass transfer coefficient in a binary system	lbs/hr ft²
G_e =	Mass Velocity of the fluid calculated on the cross section of the empty vessel	lbs/hr/ft²
$= \frac{F}{S}$	where F = volumetric flow rate of the liquid, and S = the cross sectional of the adsorber	ft³/hr ft²
a =	Surface area	ft² (of transfer area) / ft³ (of bed)
D_L =	Self Diffusion Rates	ft²/hr
μ =	Viscosity	lbs/ft.hr.
g =	Neutonian Constant	32.17 lbs (ft)/ lbs (sec)²
ρ =	Density of the fluid	lbs/ft³
ΔP =	Pressure drop	lbs/in²
H =	Height	ft
f_m =	Friction factor, a function of N_{R_e}, (modified Reynolds Number) Fig. 5-64, Perry's Handbook, page 5-51	dimensionless
D_p =	Average particle diameter defined as the diameter of a sphere of the same volume as the particle	ft
N =	Exponent, a function of the modified Reynold's Number N_{R_e}, Fig. 6-64, Perry's Handbook, page 5-51	dimensionless
Q_s =	Shape factor of the solid defined as the quotient of the area of a sphere equivalent to the value of the particle divided by the actual surface of the particle	dimensionless

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Design Calculations Equations

$$HETU = \left({}^{F}\rho/S \;/\; k_x a \right) = \frac{G_o}{k_x a} \text{ (where } G_o = {}^{F}\rho/S)$$

G_o can be determined by knowing the fluid properties and the size on the adsorber.

k_x can be calculated from "Transport Phenomena"

$J_H = 0.91\ R_e^{-0.51}\ Q_s \qquad (R_e < 50)$

$J_H = 0.91\ R_e^{-0.51}\ Q_s$

$J_D = J_H$ (By definition)

$$J_D = \frac{k_x}{cV}\left(\frac{\mu}{\rho D_L}\right)^{2/3}$$

$G_o = cV$ where

c = total molar concentration

V = volume

$$\frac{\mu}{\rho D_L} = S_c$$

$$\therefore J_D = \frac{k_x (S_c)^{2/3}}{G_o}$$

$$\text{(Substituting } J_D = J_H) \frac{k_x (S_c)^{2/3}}{G_o} = 0.91\ R_e^{-0.51}\ Q_s =$$

$$k_x = 0.91\ R_e^{-0.51}\ Q_s\ G_o\ (S_c)^{-2/3}$$

a is calculated from physical data on the adsorbents

$$a = \frac{6\ (1\text{-}E)}{Q_s\ D_p}$$

E is calculated from the following

$P_d = D_c(1\text{-}I_p)$

$P_d(1\text{-}E) = D_B$

$D_c(1\text{-}I_p)\ (1\text{-}E) = D_B$

$$E = \frac{P_d - D_B}{P_d}$$

Now G_o, k_x, and (a) can be calculated. Therefore the HETU can be calculated.

Since the total number of ETU is determined experimentally (depending on the degree of purification desired), the total height can be figured.

H = HETU x ETU

Typical Scale Up Example

General Data

Aroclor

Viscosity (μ) of Aroclor 1242 @ 70°C = 6.08 cp or 14.4 lbs/ft hr

Density (ρ) of Aroclor 1242 @ 70°C = 1.35 g/cc or 84.5 lbs/ft^3

Porocel

I_p (internal porosity)	0.50 (50%)
D_B (bulk density)	56 lbs/ft^3
D_c (crystalline density)	206 lbs/ft^3
Q_s	0.50
D_p 20/60 mesh Mean Equivalent Particle Diameter	0.0019 ft
30/80 mesh Mean Equivalent Particle Diameter	0.0014 ft

General

Newtonian Constant g = 32.2 lbs (ft)/lbs (sec)^2

Diffusion Rate (D_L) = 3.1×10^{-6} ft^2/hr

Laboratory Conditions for Refinement

Treatment temperature °C = 70

Bed Packing 20/60 mesh Porocel $D_p(ft) = 0.0019$

Column diameter (inches) = 1.62

Column Height (inches) = 60

Bed Weight (grams) = 1850

Bed Volume (cc) = 2050

Bed Density (B_D) gm/cc = 0.90

Flow Rate (cc/min) = 30

$G_o = F\rho/S$

$G_o = 380$ lbs/hr ft^2

$$R_e = \frac{G_o D_p}{\mu} \text{ (Reynold's Number)}$$

$R_e = 0.053$

$D_L = 8 \times 10^{-7}$ cm^2/sec

or

3.1×10^{-6} ft^2/hr

$$S_c = \frac{\mu}{\rho D_L} \text{ Schmidt Number}$$

$S_c = 5.5 \times 10^4$

$k_x = 0.91\ R_e^{-0.51}\ Q_s G_o\ (S_c)^{-2/3}$

$k_x = 0.485$

$$E = \frac{P_d - D_B}{P_d}$$

$P_d = D_c\ (1\text{-}I_p)$

$P_d = 206\ (1\text{-}.5)$

$= 103$

$$E = \frac{103 - 56}{103}$$

$E = 0.456$

$$a = \frac{6(1\text{-}E)}{D_p Q_s}$$

$a = 3436$ ft^2

$$HETU = \frac{G_o}{k_x a}$$

$$= \frac{380}{0.485\ (3436)}$$

$= 0.228$ ft

Since the total height of the laboratory system was 60 inches or 5 feet then, the total number of transfer units (ETU) is

$$ETU = \frac{H}{HETU}$$

$$= \frac{5.0}{0.228}$$

$= 22.0$

Since the laboratory system contained 22 units and refined satisfactorily, any scaled up system must contain this many units or preferably more.

Assume we have a column available with the following physical dimensions that we desire to use.

Diameter of the Column (ft	= 2.25
Height (H) ft	= 12.5
Area (S) ft^2	= $\pi(2.25)^2/4$ or 4
Volume (V) ft^3	= (4 ft^2) (12.5 ft.) = 50

Aroclor Data

Treatment Temperature (°C)	= 70
Viscosity (μ)$_{at\ 70°C}$ (lbs/ft hr)	= 14.4

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Density at 70°C (lbs /ft ^{3}) (ρ) = 84.5

Porocel Data

Particle Diameter D_p (ft) 0.0019

Bed Charge (lbs) 2800

$$B_D = \frac{\text{Bed Weight}}{\text{Bed Volume}} = \frac{2800 \text{ lbs}}{50 \text{ ft}^3} (\text{lbs/ft}^3) \ 56$$

Assume we desire to refine 4.5 gpm of fluid

$$\text{Flow}(F) = 4.5 \text{ gpm} \times \frac{60 \text{ min/hr}}{7.48 \text{ gal ft}} = 36 \text{ ft}^3\text{/hr}$$

$$G_o = F\rho/S$$
$$= 760 \text{ lbs/hr/ft}^2$$
$$R_e = \frac{G_o D_p}{\mu}$$
$$= 0.100$$
$$D_L = 3.1 \times 10^{-6} \text{ ft}^2\text{/hr}$$
$$S_c = \frac{\mu}{\rho D_L}$$
$$= 5.5 \times 10^4$$
$$k_x = 0.91 \ R_e^{-0.51} \ Q_s G_o (S_c)^{-2/3}$$
$$k_x = 0.725$$
$$E = \frac{P_d - D_\beta}{P_d}$$
$$P_d = D_c(1-I_p)$$
$$P_d = 206 \ (1-.5)$$
$$P_d = 103$$
$$E = \frac{103 - 56}{103}$$
$$E = .456$$
$$a = \frac{6(1-E)}{D_p Q_s}$$
$$a = \frac{6(1-.456)}{0.0019 \ (0.5)}$$
$$a = 3435 \text{ ft}^2$$
$$\text{HETU} = \frac{G_o}{k_x a}$$
$$= \frac{760}{0.725 \ (3435)}$$
$$= 0.326 \text{ ft}$$
$$\text{ETU} = \frac{H}{\text{HETU}}$$
$$= 12.5 \text{ ft} / 0.326 \text{ ft}$$
$$= 38.3 \text{ units}$$

∴ Using the existing 12.5 ft. bed column with a fluid flow of 4.5 g.p.m. would do a very adequate refining job, since only 22 units are required to give adequate refining. The actual 38 units would probably give a slightly better quality. However the advantage in an excessive number of ETU's is the gain in operating time between shut downs to change out the bed and the turn up rate available on the column feed.

Let's assume we want to double the feed on this unit to 9 gpm.

The adsorbent properties remain the same.

The only change is

G_o which changes

$$R_e = \frac{G_o D_p}{\mu}$$
$$k_x = 0.91 \ R_e^{-0.51} \ Q_s G_o (S_c)^{-2/3}$$

Then the HETU would change $\text{HETU} = \frac{G_o}{k_x a}$

$$G_o = F\rho/S$$
$$G_o = \left(\frac{9 \times 60}{7.48}\right) 84.5/4$$
$$G_o = 1520 \text{ lbs/hr/ft}^2$$
$$R_e = G_o D_p$$
$$R_e = \frac{1520 \ (0.0019)}{14.4} = 0.13$$
$$k_x = 0.91 \ R_e^{-0.51} Q_s G_o (S_c)^{-2/3}$$
$$= 0.91 \ (.13)^{-0.51} \ (1520) \ (5.5 \times 10^4)^{-2/3} \ .5$$
$$= \frac{0.91 \quad 1520}{.36 \quad 1450}$$
$$k_x = 1.325$$
$$\text{HETU} = \frac{G_o}{k_x a}$$
$$= \frac{1520}{1.325 \quad 3435}$$
$$= 0.336$$
$$\text{ETU} = \frac{12.5}{.336}$$
$$= 37.2$$

The answer is yes, the feed can be doubled. The total ETU's (37) is well above the laboratory value of (22) for adequate refinement. One can see from the equations that the adsorptive characteristics of the column are not *dependent on the flow*. However, by the same reasoning the pressure drop is shown to be highly dependent on the flow rate. Therefore pressure drop becomes a controlling factor in the design of these units. Bed height, the adsorbent, packing, and fluid temperature are likewise important factors in the bed operation.

Calculation of Pressure Drop

Assuming the 12.5 ft. bed, packed with Porocel at a bulk density of 56 lbs/ft^3 and the fluid flowing at 4.5 gpm.

According to Leva's equation for predicting pressure drop (ΔP)

$$\Delta P = \frac{2 \ f_m \ G_o^2 \ L(1-E)^{3-N}}{144 \ D_p \ \rho \ g \ Q_s^{3-N} (E)^3}$$

where f_m = the modified friction factor as determined by the Reynold's Number R_e

L = height of the bed (ft) same as H

g = Newtonian Constant 32.17 lbs (ft)/Lbs force (sec.)^2

N = exponent function of the modified Reynold's Number R_e

G_o = Superficial Mass Velocity in lbs /sec (ft)^2

The remainder of the terms are explained in the calculation of HETU.

$$\Delta P = \frac{2(1000) \left(\frac{760}{3600}\right)^2 \ (12.5) \ (1-0.455)^{3-1}}{144 \ (0.0019) \ (84.5) \ (32.17) \ (0.5)^{3-1} \ (0.455)^3}$$

$\Delta P = 18.5$ psig

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Equipment

The equipment necessary for an operational packed bed system consists of:

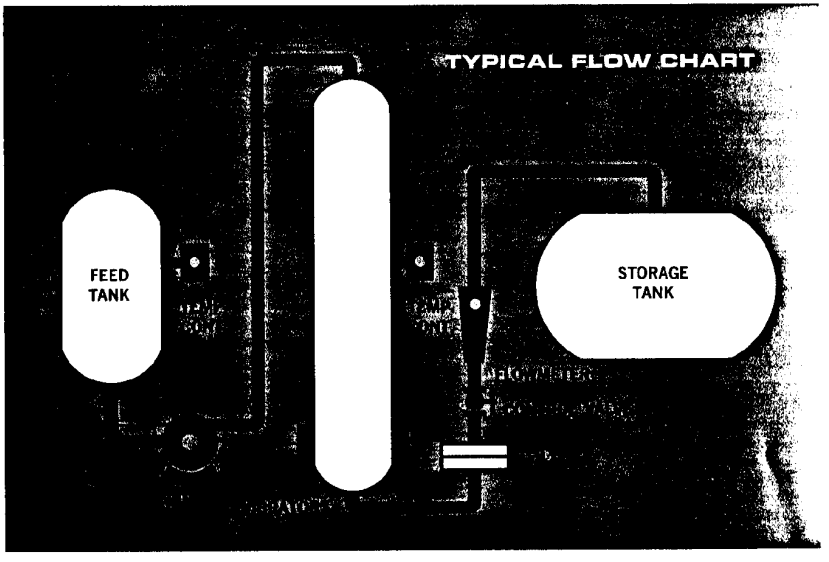
1. Feed System — a temperature control is needed on the feed for the adsorber. A positive displacement pump to yield sufficient flow at the operating pressure is necessary.

2. Adsorber — The actual adsorber shell must be a suitable material of construction such as aluminum or stainless steel to eliminate the possibility of product contamination. The surface of a stainless adsorber should be cleaned free of any rust. The top of the adsorber should be removable for ease of loading the adsorber. The bottom should be equipped with an adequate bed support to retain the granular adsorbent and yet allow the liquid to pass freely. The bottom should contain a dump port large enough to allow for removal of the bed material. The unit should be adequately traced for temperature control. A temperature indicator should be installed in the adsorption zone. Vibrators should be installed on the shell of the adsorber large enough to pack the bed to optimum density. A N_2 purge should be connected to the bottom of the adsorber and sufficient heat provided to drive off moisture in case the water content of the adsorbent is too high when the column is loaded. The normal recommended height to diameter is about 4-5 to 1 although a shorter unit could possibly be used. Less than 3 H/D is not recommended. The normal safety items for a pressurized vessel are also necessary.

3. Filter — Small in-line polishing filters are needed on the column to catch any residual adsorbent, etc., that may pass through the bed support. The filters should be on a by-pass arrangement to keep the unit on stream at all times.

4. Flow Controller — A flow controller should be placed on the effluent of the adsorber to control the fluid flow through the adsorbent.

5. Storage System — A storage tank for the finished product should be provided with sufficient capacity to keep the column on stream.

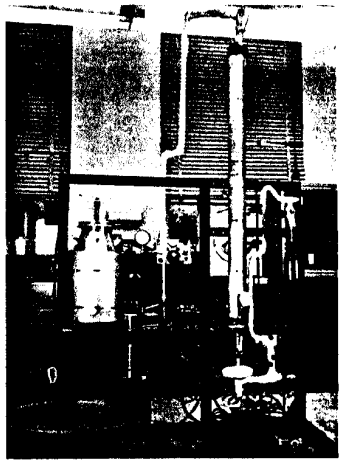


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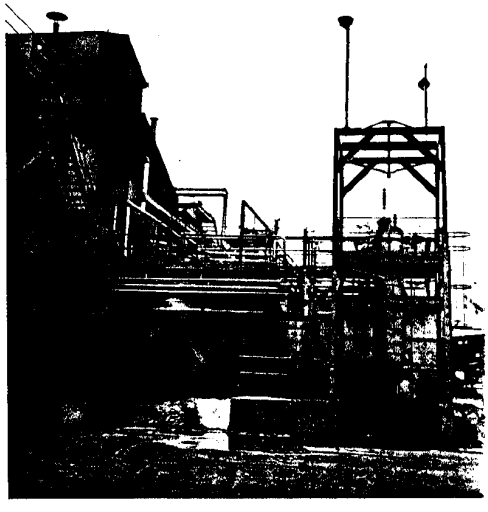
Adsorber Operation

The column, after being throughly cleaned, is filled with activated (dry) Porocel. The bed is settled to an optimum density through the use of vibrators. The bed is then flooded with the fluid and allowed to soak into the bed and displace any air which may be trapped in the adsorbent. After the bed has been allowed to soak, preferably over night at the operating temperature the forward flow should then be started. Once the quality reaches an optimum level, the bed will continue to operate at this level until it becomes saturated with adsorbatc. When the bed becomes saturated, the quality will show a sharp decrease or breakthrough. At this point the quality will drop to a considerably lower level and hold for a period of time and then drop to a completely unacceptable quality level. Once the breakthrough is obtained, the bed should be changed and the cycle started over.



Shown above is a small laboratory model capable of producing up to 50 cc/min. of refined askarel. This unit can be used to produce laboratory quantities or collect scale-up data to design any size unit up to or larger than the unit shown below.

This production model produces the highest quality refined askarel under the most economical conditions. Because of the efficiency, these units operate continuously for months on a single bed charge.



Operating Costs

Savings are realized from

1. Manpower reduction
2. More efficient adsorbent utilization
3. Continuous faster refinement
4. Less contamination
5. Less product loss
6. Reduced maintenance

The only disadvantage of the porocel column is the higher cost of the adsorbent (about 50%), but this is practically offset by the increased efficiency of the Porocel (40%).

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Actual Field Experience

The Monsanto laboratory tested a small unit collecting scale-up data for a pilot design. After a successful pilot demonstration, a production unit was brought on stream yielding continuous refinement of the Askarel (except for short intervals for changing the bed). Due to the efficiency of the system and the high quality product produced, Monsanto is adopting the system as a production standard worldwide. In addition, the system is being disclosed to those customers desiring to use the most efficient and economical method of refinement.

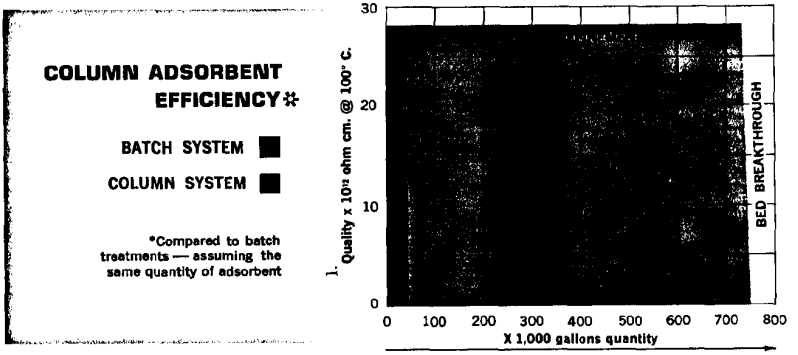
Typical Results Achieved 1.

Fluid	Resistivity (ohm cm)* x 10^9
Aroclor 1260	230,000
Aroclor 1254	130,000
Aroclor 1248	40,000
Aroclor 1242	28,000
Mixture of Aroclor 1260, TTCB, TCB, and dicyclodiepoxy carboxylate	80,000
Trichlorobenzene	18,000

*Conventional concentric electrodes, 0.1 inch spacing 500 VDC stress with 1 minute electrification, @ 100° C.

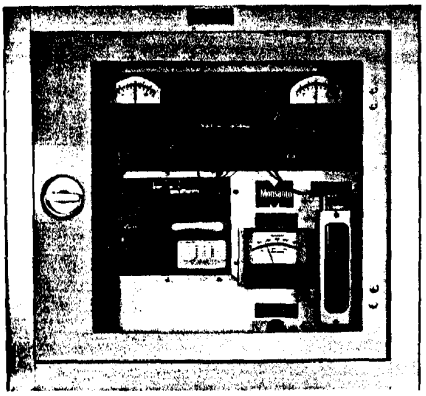
Besides the increase in quality, the column adsorber system was found to be 18 to 20 times more efficient than old contact system. The increased efficiency in earth utilization of the column system reduces manpower requirements when compared with the old batch system.

1. Results are reported as resistivity which is the reciprocal of power factor. At these quality levels, unless the new guarded electrode power factor system is used, an accurate measurement cannot be made.



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A Monsanto Power Factor System constantly monitors on-stream quality through 100 cycle power factor at 100° C. This system requires no external sampling for quality control.

Optional Equipment

The Monsanto "in line" analyzer is a very useful instrument for monitoring the product quality from the column. The unit was designed specifically for quality control on the effluent from this system. It provides a continuous monitoring of power factor and has provisions for recording if necessary. The quality of the refined material is known constantly within three minutes after refinement. The "in line" analyzer is invaluable during start up of the adsorber to determine when recycle should be stopped and the forward flow diverted to the storage system.

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