Coalescence in Fibrous Beds

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The factors affecting the efficacy of a closely packed bed of mixed cotton and a supporting fiber (Teflon, glass, Dynel) were evaluated for oil-in-water dispersions. Several other water-organic systems were also tested. Superficial velocities ranged from 0.2 to 3.5 ft./min. Successful coalescence was attained at interfacial tensions as low as 3.5 dynes/cm. Dispersed phase viscosity was varied from 1.4 to 137 centipoise. For a mixed-fiber bed with a specific ratio of fiber species, there is an optimum bed depth for best performance. High-speed cinephotomicrographic observations at 100× and up to 4,000 frames/sec. indicated that fiber wettability is not the most important factor for successful operation.

Organic chemicals must be purified during their manufacture by contacting them with immiscible solutions in a washing or extraction operation. The immiscible solution is usually aqueous. The most prominent examples are in the field of petroleum products such as gasoline and kerosene. Fuel for modern jet-powered airplanes is a particularly important product in which the presence of water is a very critical problem (*7*). The reverse situation, in which very fine droplets of an oil phase are dispersed in a large amount of water, is of considerable interest in the prevention of contamination of rivers, harbors, and beaches.

If a water phase is dispersed in an organic liquid by any of the efficient turbulence creating mixers presently in use, an emulsion will be formed. If both liquids are pure (no surfactants present), the emulsion will be of a temporary nature and both phases may be recovered by a simple settling operation. If a strong surfactant is present, the emulsion may be a permanent one requiring special methods to break it.

Two types of temporary emulsions are recognized (*12*). The first (primary) is characterized by a drop size of the order of 100 μ and will separate readily in a few seconds or minutes by simple settling. If one or both of the two liquids involved contains polar compounds, one or both of the two layers which result from the settling operation will be clouded with a fine mist or fog of extremely fine droplets of the opposite phase (*1*). Such fogged layers are called secondary emulsions and consist of untold billions of droplets of submicron size suspended in a field of the opposite phase. These secondary emulsions cannot be settled clear for many minutes or hours. Coalescence of these tiny droplets into large ones is necessary if the emulsion is to be broken and separation attained.

Passage of an emulsion through a fibrous bed will often cause coalescence and facilitate separation (*12*); sometimes such beds are dependable and sometimes not. The mechanism of their operation is shrouded in vagueness and conjecture. No accepted theory exists on how they accomplish the coalescence of the submicron droplets into large ones of manageable size. Were one available, it would facilitate design and aid selection of materials of construction of such devices. The present work is concerned with checking the mass of seemingly conflicting testimony regarding the performance of such fibrous beds and, by establishing the role of the primary variables involved, to formulate a set of workable relationships.

We are not concerned herein with those fibrous beds or screens which act as a hydrophobic barrier, or filter, upon the front face of which the drops may be held to coalesce (*8*).

PREVIOUS WORK

Based upon gross performance observations on both small test cells and full size industrial equipment, the generalizations listed below may be tentatively accepted. Experiments can be devised and carried out in such a manner as to either confirm or deny nearly all of them.

1. The fibrous bed must be closely packed and possess a high ratio of surface area to volume (*12, 15*).
2. The size of the capillary openings must be relatively large. Note that 1 and 2 are consistent if very fine fibers are used (*12*).
3. The fibers must be preferentially wetted by the dispersed phase (*9, 12, 15*). Several authors (*3, 4, 8, 10, 11*) have indicated that this is not universally true.
4. The flow rate must be above a certain minimum but below a certain maximum. Superficial velocities between 0.25 and 1.0 ft./min. seem satisfactory (*2, 12, 15*).
5. Higher temperatures promote the operation (*2, 12*).
6. Thin beds will separate coarse emulsions (50-μ droplets) but a bed thickness of several inches is required to coalesce secondary emulsions of submicron drops (*2*).
7. A high interfacial tension system is more easily coalesced than is one of low interfacial tension (*6, 13*).
8. Surfactants, dirt, and high viscosity all tend to prevent coalescence (*13*).
9. A potential energy barrier, which may be electrical or mechanical or differential wetting, can prevent the operation (*1*).
10. Large drops form and grow on the leading surface.
11. Large drops wander through the bed in an irregular path to the down stream surface.
12. Large drops appear at, grow, and leave the downstream surface. Small drops are often formed at this point by breaking of the usual connecting thread of drop liquid.
13. It has been observed that while the fibers must be hydrophilic to the droplets, they must not be too hydrophilic. An intermediate value appears best (*15*).

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MECHANISMS

Several mechanisms of operation have been devised to explain the manner of operation by which such beds promote coalescence of micron and submicron size drops within the fibrous structure.

Inertial Impaction

If the particles have the same relative mass as the field phase, they will usually follow the fluid streamlines around any obstruction in the stream. If the flow is turbulent and if vortices occur, the drops can be brought to a fluid interface even though the density difference be low. Redmon (*8*) has offered the most authoritative opinion, that low density difference systems are extremely difficult to coalesce. If the drop density is less than that of the field liquid, the inertial impaction mechanism should act to prevent drops approaching the fiber or other drop surface. Such a phenomenon is not universal.

Direct Interception

If a particle has a finite diameter, it will tend to touch the cylindrical fiber when the distance $D_p/2$ from the collector surface is reached. Rose (*10*) and Vinson (*14*) have treated this phenomenon for cylindrical fibers or wires. It has not been fully treated for spheres, but the same relationships should hold.

Brownian Movement

Particles in the submicron size range can reach each other as well as other wetted surfaces due to Brownian motion (*14, 15*). Droplets above a very few micron diameter are not susceptible to this action.

Electrostatic Movement

Particles which carry an electrostatic charge can be attracted to those of opposite charge. The alternating voltage field used in some commercial settlers promotes such action. Like Brownian movement, it is very effective on the very smallest particles.

Rupture in Capillaries

It has been postulated that when the emulsion is passed through a fibrous bed, there is a tendency for the micron size droplets of the dispersed phase to be forced together as they move through the passages. Film thinning would then occur and the drops would coalesce together while moving. Photomicrographic studies have shown that little or no coalescences occur between two freely moving droplets which contact each other as they are carried through the bed (*4, 11*). One of the drops must be held in the bed so that other drops may come to it for film thinning and rupture to occur. It must be borne in mind that closely packed fibrous beds are about 90% free space and that the size of the smallest moving drops is far less than the diameter of the fibers or of drops held stationary in the bed.

EXPERIMENT

Conflicting published information, much of which was qualitative and speculative in nature, indicated the necessity of carrying out a series of experiments designed to select a fibrous bed which could be used to coalesce a secondary emulsion of oil dispersed in water. While the primary objective was concerned with O/W emulsions, experiments on W/O dispersions were also conducted to check the validity of a number of unsupported ideas. The effects of surfactants, bed properties, fluid properties, and operating variables were determined.

Equipment

The apparatus used is shown diagrammatically in Figure 1. After selecting the fibers to be used, a bed was prepared and supported in a hollow brass cylinder 2.75 in. long with an I.D. of 1.50 in. The cylinder had a stainless steel screen soldered to one end and was attached to a ¼-in. brass mounting plate. This screen was made of 0.11-in. wire and had ⅛-in. openings. The bed was secured by a circular stainless steel screen, which was inserted through the open end of the cylinder and held in place by an aluminum bolt that passed through both screens and the fibrous bed. The movable screen was made of 0.0758-in. wire and had 1/16-in. openings.

The fibrous bed support was mounted horizontally between two 2-in. Pyrex glass pipe tees. The outlets of the tees were sealed by ¼-in. brass plates with ⅛-in. neoprene gasketing. The plates over the horizontal outlets contained openings through which the liquid could enter or leave. Also, there were openings in the upper outlet plates to allow pressure measurements to be made and to aid in the drawing off of the coalesced dispersed phase.

The emulsion was continuously forced through the system by a 1,725 rev./min. stainless steel gear pump. The piping to and from the tees was ¼-in. O.D., 0.03-in. wall, copper refrigeration tubing. A by-pass line of ¼-in. brass pipe was installed between the pump and the tees to aid in controlling the liquid flow rate to the tees. All the valves and fittings were brass. The flow rate was determined with a Fisher and Porter rotameter (model 6012A2786B1).

The survey tests were made in a similar apparatus but the direction of flow was vertical; downward if the dispersed phase was heavier and upward if it was lighter than the continuous phase.

Operating Procedure

After the fibrous bed was installed in the system, the pump was turned on and the emulsion was allowed to flow through the by-pass line until steady operation was reached. The valve which controlled the flow rate to the tees was then opened and the emulsion passed through the coalescing system. An experimental run, at one flow rate reading on the rotameter, lasted 15 min. The flow rate, pressure gauge reading, temperature, coalescence, and redispersion effects were observed for each run. The flow rate was then increased to another value. Data were taken for a velocity range from 0.1292 to 3.52 ft./min. superficial velocity.

Preparation of the Emulsion

The emulsion was prepared by mixing the petroleum fraction and distilled water in a stainless steel tank. The concentration of the dispersed phase in the emulsion was in the 2 to 5% by volume range. A 1-5/16-in. stainless steel turbine impeller was used to agitate the emulsion. The emulsion was pumped from the reservoir through the system and then returned to the reservoir.

Fibers

The fibers used were medical cotton, Dynel, glass, polyethylene, polypropylene, and Teflon; the size and density of each are listed in Table 1. Mixed fibrous beds of cotton-glass, cotton-Teflon, and cotton-Dynel were tested. The Teflon fibers used in the mixed beds were very coarse and were somewhat

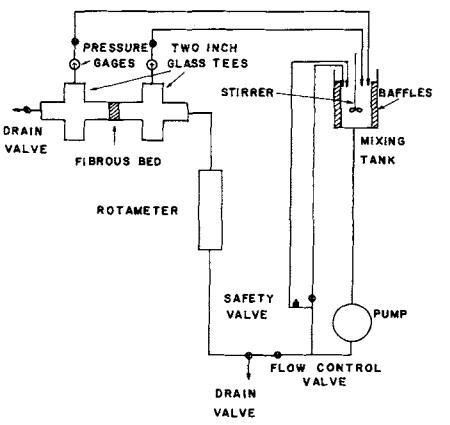

Fig. 1. Equipment diagram.

rigid cylinders of about 100μ in diameter. A fibrous bed of medical cotton can be made to coalesce almost any kind of phase from any other phase. Exceptions are the very low normal interfacial tension systems as well as O/W emulsions in which the interfacial tension has been lowered to less than 3 dynes/cm. by the addition of certain surfactants. But cotton fibers have little rigidity and will collapse (8) at relatively low pressure drops. Therefore mixed-fiber beds of cotton-glass, cotton-Dynel, and cotton-Teflon were used to take advantage of the good qualities of cotton and the supporting properties of other materials.

Preparation of Mixed-Fiber Beds

The mixed-fiber beds were prepared by two methods.

Cotton-Glass Fiber Bed: The fibers were precut into ½-in. lengths to aid in their handling. The appropriate amount of each fiber was then weighed out. The correct weight of each fiber was specified by the particular weight ratio $R_f = S/(S + C)$ desired. For our purposes, S refers to the weight of the supporting fiber and C refers to the weight of the cotton fiber used. The fibers were then dispersed in separate beakers of distilled water. A suction apparatus was attached to the bed support and the fibers were poured into the support in alternating layers, which were approximately ⅛-in thick.

The suction was then turned on to dry the fibers, after which the circular stainless screen was inserted and the bed was compressed to the desired thickness. The bed and support were then allowed to soak in the pure dispersed phase for approximately 12 hr. before an experimental run was begun.

Cotton-Dynel Fiber Bed: A weighed amount of cotton and Dynel was layed out in the form of layers of cotton and Dynel and then cut into ½-in. strips. All the cut fibers were dispersed in distilled water and thoroughly mixed. The dispersed fibers were taken out and squeezed to remove most of the moisture contained in them. They were then packed into the hollow brass cylinder and allowed to dry. This method of making a bed was termed as a *random-packed bed*. The dried fibers were then compressed to the desired thickness and allowed to soak in the pure dispersed phase for approximately 12 hr. It should be noted that the bed density was maintained constant by varying the total weight of fibers used as the bed depth was changed.

Determination of Coalescence

The degree of coalescence was determined visually as a function of the cloudiness in the downstream tee and classified as complete, partial, or incomplete. For the situation where normal visual determination of coalescence was doubtful, a light beam was passed through the downstream tee. If coalescence was complete, the light beam passed through the tee with no reflection. If the coalescence was incomplete, however, the light beam was reflected by the micron sized particles of the dispersed phase and a haze was visible in the tee. The changes from complete to partial and from partial to incomplete coalescence were gradual, as would be expected, since the increments in velocity were small.

It was observed that small individual droplets emerged from the fibrous bed and were carried through to the downstream tee by the continuous phase before they could settle out. These droplets underwent some degree of coalescence in the bed, but were not large enough to attain complete separation. This phenomenon was referred to as *redispersion* and was classified as none, slight, or heavy. The velocity at which redispersion occurs is called the *critical separation velocity*.

TABLE 1. FIBER PROPERTIES

Material	Fiber diameter, μ	Material density, g./cc.
Cotton	14.05	1.58
Dynel	43.5	1.31
Glass	8.85	2.50
Polyethylene	46.5	0.92
Polypropylene	45.0	0.90
Teflon	20.0	2.3

TABLE 2. PHYSICAL PROPERTIES OF LIQUIDS

Liquid	Density, g./cc.	Viscosity, centipoise	Interfacial tension, dynes/cm.	Temperature, °C.
Benzene	0.879	0.652	35.0	20.0
n-Butyl benzoate	1.005	2.79	31.0	25.0
Carbon tetrachloride	1.574	0.883	33.2	26.5
Chloroform	1.485	0.542	32.8	25.0
Cyclohexanol	0.962	68.0	3.92	20.0
Isobutanol	0.802	4.70	2.1	20.0
Kerosene	0.8006	1.483	47.0	25.0
Nitrobenzene	1.2007	1.915	23.7	22.5
No. 10 Base oil	0.80879	1.3774	45.0	28.0
LF-1584 Oil	0.8996	137.5375	52.2	28.0
LF-1584 Oil—10% No. 10 Base oil—90%	0.81746	1.7321	45.3	28.0
LF-1584 Oil—20% No. 10 Base oil—80%	0.82759	2.3803	46.4	28.0
LF-1584 Oil—30% No. 10 Base oil—70%	0.8378	2.9911	46.8	28.0
LF-1584 Oil—40% No. 10 Base oil—60%	0.84868	3.925	48.3	28.0
LF-1584 Oil—60% No. 10 Base oil—40%	0.869	11.169	48.9	28.0
LF-1584 Oil—80% No. 10 Base oil—20%	0.88863	28.61	49.8	28.0

Materials

The materials used were No. 10 base oil and LF-1584 oil supplied by the American Oil Company. The other organic liquids and their physical properties are listed in Table 2.

The surfactants used were Tergitol TMN (trimethylnonyl ether of polypropylene glycol, 90% plus, $\sigma = 25.9$ dynes/cm.) and Sarkosyl-O (oleyl sarcosine, molecular weight 345 to 360, 94% plus, specific gravity 0.948).

All the fibers used are commercially available and have uniform properties (Table 1). Medical cotton was purchased at the local pharmacy.

Physical Properties

The density and viscosity of the kerosene, No. 10 base oil, LF-1584 and the various mixtures of the latter two were measured with a specific gravity bottle and Ostwald-Cannon-Fenske viscometers, respectively. The surface tension and interfacial tension of all the liquids were measured with a Cenco DuNouy ring tensiometer. The density and viscosity of the other organic liquids were obtained from handbooks, because they were certified pure. All physical properties are listed in Table 2.

DISCUSSION

Preliminary Survey Tests

Nine water-organic systems were used with density differences ranging from 0.2 to 0.57 g./cc. and interfacial tension from 2.1 to 47 dynes/cm. Six fibers were used with mean diameter varying from 8.85 to 46.5 μ. The systems used and their physical properties are presented in Table 2. Fiber properties are presented in Table 1, and a summary of runs is presented in Table 3.

Secondary emulsions of organics in water and water in organics were prepared and pumped through the apparatus shown in Figure 1. The emulsion was forced through the fibrous bed, prewet by the dispersed phase, to determine if any coalescence took place, and the coalescence effect was noted visually.

The results of these preliminary tests are in Table 3. Complete coalescence (C) was deemed to occur if the effluent continuous phase was clear and free from haze as seen by side lighting with a narrow light beam. Large drops of the dispersed phase grew and left the downstream surface of the bed. If the exit field fluid was

TABLE 3. PRELIMINARY TESTS OF FIBERS AND SYSTEMS

System	Cotton	Dynel	Glass	P-E	PP	Teflon
Benzene-water	C	N	C	N	—	P
Butyl benzoate-water	C	P	P	P	—	P
Water-carbon tetrachloride	C	P	C	P	—	C
Chloroform-water	P	N	N	N	—	N
Clyclohexanol-water	N	—	N	N	—	—
Isobutanol-water	N	N	N	N	—	N
Water-isobutanol	C	P	C	P	—	P
Water-kerosene	C	P	C	P	P	C
Nitrobenzene-water	N	—	N	N	—	N

C = Complete coalescence at all flow rates. *N* = Very slight or no coalescence. *P* = Partial coalescence leaving some haze in suspensions. In some cases there was redispersion.

slightly fogged or if redispersion of the coalesced material into tiny droplets of less than 1 mm. occurred, the coalescence was termed partial (*P*). If the effluent continuous phase was quite fogged, it was deemed that no real coalescence was effected (*N*) by the fibrous bed system. The effluent stream was not analyzed for dispersed phase content because this concentration is dependent on the settling characteristics of the downstream chamber.

It will be noted that medical cotton is the best fiber of all, closely followed by glass fibrous beds. Dynel, polyethylene, and polypropylene fibers were about 45 μ in diameter and could not be packed closely enough to make an effective bed. Teflon fibers of 20 μ diameter were effective coalescers when water was the dispersed phase and the interfacial tension was high. Teflon beds can also act as a dispersion device at the flow rates used. Drops of large size cannot be held with sufficient tenacity to resist the hydrodynamic pressure on them. Chloroform and nitrobenzene were poorly coalesced by any of the fibrous beds. These are systems of high interfacial tension but also of a high degree of polarity. It is believed that their highly polar nature causes a resistance to coalescence. They are easily dispersed and will cause a secondary emulsion to form readily. Emulsions of cyclohexanol in water and isobutanol in water could not be broken by any of the beds. Both systems exhibit a very low interfacial tension. Both cotton and glass fibrous beds coalesced water from an isobutanol field. It would seem that preferential wetting is an all compelling factor for this low (2.1 dynes/cm.) interfacial tension case. It must be noted here that these are *natural* low interfacial tensions systems as contrasted with systems in which a low static interfacial tension is attained through the use of a surface-active agent.

The difference in density of the two mutually saturated liquid phases was brought to a low value in the *n*-butyl benzoate vs. water system. It should be almost impossible to coalesce such a system. Inertial impaction and direct interception should be inoperative, since the micron size droplets can cross streamlines with great difficulty. But cotton beds were very effective and the other fibers were partly so. A superficial velocity of 3 ft./min. of water in a 2-in. tube will result in a pipe Reynolds number of about 700. The same velocity past a 7-μ diameter drop will result in a drop Reynolds number of about 0.10. If the fibers be about 15 μ in diameter, the Reynolds number in the packed bed (90% free space) will be about 0.2. This would indicate that any turbulence should be of a very low order and an absence of vortices behind drops or fibers. However microscopic observations showed that this was not the case. Turbulence in the bed is quite high and vortex shedding may be seen to be plentiful at such flow rates in such beds. It is believed that this is the main reason why the beds were effective on the *n*-butyl benzoate–water system.

The survey data indicate that several phenomena may be operating simultaneously to promote coalescence of micron and submicron size drops to a drop size of at least 1 mm. Billions of the former are required to make one of the latter. Microscopic observations have shown that small (about 5 μ) drops do not coalesce while moving together in a moving stream. They are not easily pushed together, so the sequence of events (approach, contact, film thinning, rupture, hole expansion) of coalescence can occur. All observed coalescences occurred only if one or more drops were held at a position in the bed so that other drops could come to them to combine. The mechanism of operation of such beds therefore must be explained by considering how drops may be held in the bed and how they can leave.

Effect of Fibers

The degree of coalescence effected by a given fibrous bed is related to the number of drops which adhere to the fibers and to the length of time the drops remain on the fibers. A droplet that is held on a fiber for a long period of time will undergo a greater number of collisions with free dispersed phase droplets and, as a result, the degree of coalescence should increase. The superior coalescing ability of the cotton indicated that the petroleum fraction droplets were held more securely by the cotton fibers than by the Teflon or glass wool fibers. The best coalescence achieved, among all the fibers tried, was by cotton and this is shown in the systems: nitrobenzene–water, butyl benzoate–water, benzene-water, water-carbon tetrachloride, water-isobutanol, and water–kerosene.

The photomicrographic investigation showed that in most cases, the droplets which adhered to the cotton fibers did not actually wet the fibers. There was a zero contact angle between the droplet and the fiber. The droplet fillet, characteristic of wetted adherence, was not observed. Thus, it appears that preferential wetting was not the controlling factor which made cotton a more efficient coalescer than the other fibers.

These experimental findings may be justified if the relative roughness of the fibers is considered. The natural, ribbonlike cotton fibers do not have smooth surfaces, but possess many depressions and obstructions, which increase the fibers ability to capture and to hold the droplets of the dispersed phase. This is not true for the other smoother synthetic fibers. As a result, the cotton is able to provide more complete coalescence for a longer period of time and, theoretically, it should continue to effect coalescence indefinitely.

However, cotton used by itself has a disadvantage in that it tends to pack very tightly, thus increasing the pressure drop across the bed. In order to minimize this factor mixed-fiber beds, cotton-glass wool and cotton-Dynel, were used. The glass wool and Dynel were used as supporting fibers. Dynel, in particular, is a crinkled, resilient fiber and, therefore forms an excellent supporting medium.

Effect of Bed Depth

Bed depths varying from 0.25 to 1.25 in., increasing in increments of 0.25 in., were investigated while keeping the R_f ratio constant. The results indicated that as the bed depth increased the degree of coalescence also increased. This agrees well with the findings of other workers.

However, there is a limitation as to just how deep the bed can be. It is true that because of a longer residence time in the deeper bed the coalescence is better, but there is also a high-pressure drop across it and this results in the redispersion of the coalesced drops. It was observed that redispersion always took place before the onset of partial coalescence. When redispersion occurs some of the

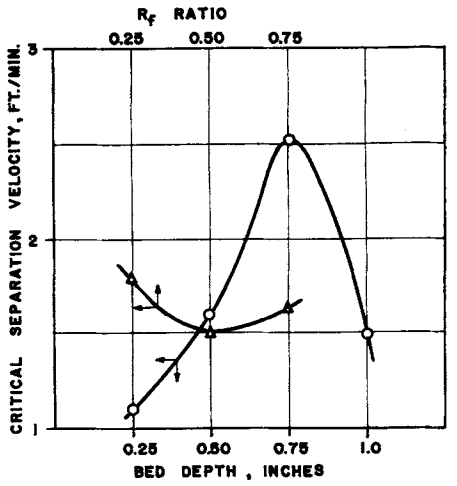

Fig. 2. Critical separation velocity vs. bed depth and R_f ratio; cotton and glass fibers; No. 10 base oil.

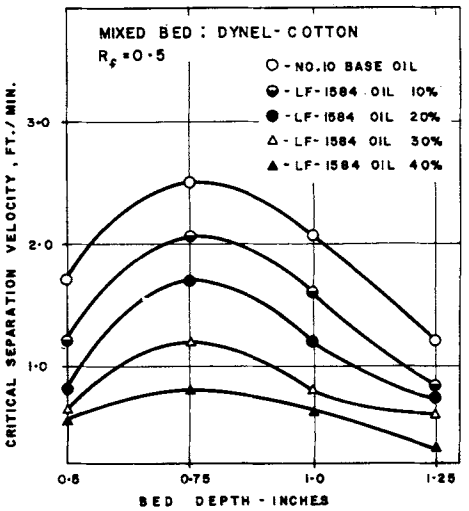

Fig. 3. Critical separation velocity vs. bed depth for cotton and Dynel fibers.

drops emerging from the bed are too small to settle out before they pass through the downstream tee. This phenomenon will occur if a drop does not remain on a fiber long enough to undergo a sufficient degree of coalescence or if the large drops formed in the bed are broken up as they move through the narrow channels formed by the fibers in the bed.

After a small droplet has adhered to a fiber in the packed bed, it can increase in size if it contacts and coalesces with other droplets being carried through the bed by the continuous phase. Photomicrographic studies have shown that a drop will break away from the fiber after it has attained a certain size. This breakaway is caused by the hydrodynamic pressure on the drop. If the superficial velocity of the emulsion is high enough, a held drop may be carried away before it has reached the size necessary to enable it to settle out after leaving the packed fibrous bed.

For a mixed-fiber bed with a specified R_f ratio, there is a superficial velocity (critical separation velocity) below which a packed bed of a given depth must be operated so that complete separation (complete coalescence and no redispersion) is achieved. The optimum bed depth for an R_f ratio of 0.5 was determined to be 0.75 in. This is shown in Figures 2 and 3.

Effect of Viscosity

The LF-1584 oil and the No. 10 base oil were mixed in various proportions to give a viscosity range of 1.3774 to 137 centipoise. From the plot of the critical separation velocity versus viscosity, at a constant R_f of 0.5 and bed depth of 0.75 in. (Figure 4), it is observed that at velocities greater than 2.5 ft./min. and less than 0.13 ft./min. there is no effect of the viscosity on the critical separation velocity, that is, it is asymptotic at these two values.

Effect of Surfactants

Experiments were also conducted to study the effects of surface-active agents on coalescence. Tergitol TMN was dissolved in distilled water in various concentrations to give interfacial tension values of 30.3, 17.4, and 11.4 dynes/cm. For the higher interfacial tension value (30.3 dynes/cm.) partial coalescence was observed at all velocities, while for the interfacial tension values of 17.4 and 11.4 dynes/cm. incomplete coalescence was observed at all velocities.

On the other hand, when Sarkosyl-O was dissolved in the No. 10 base oil in various concentrations to give interfacial tension values of 31.05, 12.18, 3.52, and 2.33 dynes/cm., a different effect was observed. For the values 31.05, 12.18, and 3.52 dynes/cm. complete coalescence was observed at all velocities, whereas for the 2.33 dynes/cm. value, incomplete coalescence was noticed at all velocities. The lowest interfacial tension value that could be obtained was about 2.33 dynes/cm. The interfacial tension tests were carried out with a Cenco DuNouy Tensiometer No. 70545.

It was also found that some of the Sarkosyl-O was soluble in the continuous phase and that some of the Tergitol TMN was soluble in the oil phase.

The model used to explain this phenomenon of incomplete coalescence for a water-soluble surfactant and the complete coalescence for the oil-soluble surfactant, is as follows:

Consider a drop about to coalesce with another drop. In order that they may coalesce there must be rupture of the separating film of water present. With the water-soluble surfactant, which lowers the interfacial tension to below 20 dynes/cm., the interfacial film toughens and thus makes coalescence almost impossible.

On the other hand, for the oil-soluble surfactant, most of the surfactant is present inside the oil droplets and there is, therefore, little toughening of the separating film of water, thereby permitting easy coalescence of the drops.

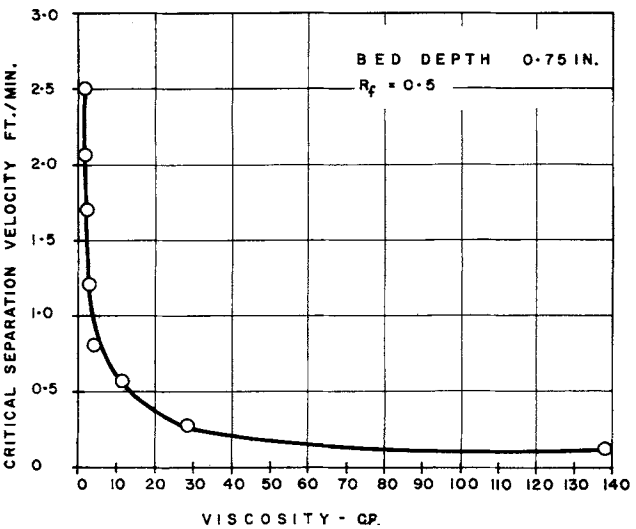

Fig. 4. Critical separation velocity vs. viscosity of dispersed phase.

No noticeable effect of the surfactants, Tergitol TMN and Sarkosyl-O on the efficiency of the bed to coalescence was observed. Tergitol TMN dissolved in water was run first and then Sarkosyl-O dissolved in No. 10 base oil was run next. The bed was not cleaned with distilled water between the water-soluble surfactant runs. The bed used for the runs with the surfactants was used for a month and runs were made almost every day for periods of about 4 hr.

In order to determine the durability of the mixed-fiber bed (cotton-glass wool), the bed operation was carried out in 8-hr. intervals for a total time in excess of 70 hr. and the degree of coalescence achieved did not decrease.

CONCLUSIONS

1. Preliminary experiments indicated that more than one mechanism takes place during coalescence.
2. Coalescence of two systems, in which the dispersed phase was lighter than the field phase, was achieved with cotton, glass wool, and Teflon fibrous beds. Dynel and polyethylene did not yield complete coalescence.
3. Electrostatic phenomena sometimes occur and may aid in the coalescence of the dispersed phase by promoting attraction and adhesion between the droplet and the fiber surface.
4. Foam formation at the exit of a fibrous bed has been found to be dependent on bed density. Qualitatively, the tendency to form foam varies inversely with the density of the packed fibrous bed.
5. As the fiber diameter is decreased, more complete coalescence is obtained.
6. For each combination of system and fibrous bed a minimum bed density is required to achieve complete coalescence of the system.
7. Coalescence of the dispersed phase into large drops was found to occur preferentially at certain fixed points in a given bed.
8. Mixed-fiber beds, composed of cotton and glass wool and cotton and Dynel, were found to be efficient coalescing mediums for secondary emulsions.
9. These mixed-fiber beds are very durable and will continue to effect complete coalescence over long periods of operation. Discontinuous operation does not hamper the bed's efficiency.
10. It appears that the droplets of the dispersed phase did not undergo wetted adherence when they were in contact with the bed fibers. There was a zero contact angle between the droplets and the fibers.
11. The superior coalescing quality of cotton fibers is probably due to the relative roughness of the fibers. The surfaces of the cotton fibers contain many depressions and obstructions, which aid in the capture and adherence of the dispersed phase droplets.
12. The degree of separation achieved by the mixed-fiber bed device was found to be a function of the coalescence effect and the amount of redispersion. Redispersion was the controlling factor in the determination of the critical separation velocity for both the bed depth and the R_f ratio investigations.
13. For a mixed-fiber bed with a given R_f ratio, there is an optimum bed depth to be used if complete separation is desired. A bed depth of 0.75 in. was the optimum for the mixed-fiber beds used, with an R_f ratio of 0.5.
14. The maximum coalescing velocity increased with increasing bed depth. For a mixed-fiber bed with an R_f of 0.5 and for the No. 10 base oil, the coalescing velocity increased from 3.2 ft./min. for a bed depth of 0.50 in. to over 3.5 ft./min. for a 1.25-in. bed.
15. The superficial velocity at which redispersion occurred reached an optimum at a bed depth of 0.75 in. for a mixed-fiber bed with an R_f ratio of 0.50. The critical separation velocity was 2.5 ft./min. for this bed depth and R_f ratio.
16. Complete coalescence was observed at velocities as low as 0.1292 ft./min. This is because at low velocities Brownian movement is predominant and as a result of this the particles tend to migrate from the flow lines, thereby increasing the frequency with which they collide with one another and the fibers.
17. Very low and high viscosities of the pure dispersed phase do not have any effect on coalescence. The critical separation velocity for viscosities less than 1 centipoise, is greater than 2.5 ft./min. and for viscosities greater than 130 centipoise, is less than 0.13 ft./min. (the lowest value that can be read off the rotameter used).
18. Surfactants Tergitol TMN and Sarkosyl-O do not affect the properties of the bed or of the fibers in it.
19. Complete coalescence was achieved with the oil-soluble surfactant (Sarkosyl-O) for interfacial tension values as low as 3.52 dynes/cm. At an interfacial tension value of 2.33 dynes/cm., incomplete coalescence was observed. There is, therefore, a critical value of the interfacial tension between 2.33 and 3.52 dynes/cm., where the transition from incomplete to complete coalescence takes place. This critical value is difficult to determine because, first, it is difficult to make a mixture of the surfactant and the oil to give a series of values between 2.33 and 3.52 dynes/cm. (extremely small quantities of the surfactant drop the value of the interfacial tension to small values); and second, the accuracy of measurement of the interfacial tension at such low values is questionable.
20. Incomplete coalescence was observed at interfacial tension values of less than 20 dynes/cm. with the water soluble surfactant Tergitol TMN. At interfacial tension values of 30 dynes/cm. partial coalescence was observed.

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LITERATURE CITED

1. Broughton, G., *Ind. Eng. Chem.*, **49**, 919 (1953).
2. Burtis, T. A., and C. G. Kirkbride, *Trans. Am. Inst. Chem. Engrs.*, **42**, 413 (1946).
3. Farley, R., and F. H. H. Valentin, paper presented at meeting of A.I.Ch.E.-Inst. Chem. Engrs. (June, 1965).
4. Gudesen, R. C., M.S. thesis, Illinois Inst. Technol., Chicago (June, 1964).
5. Hayes, J. G., L. A. Hays, and H. S. Wood, *Chem. Eng. Progr.*, **45**, 235 (1949).
6. Jordan, G. V., *Trans. Am. Soc. Mech. Engrs.*, 394 (April, 1955).
7. Langdon, W. H., E. G. Fochtman, L. L. Stark, and M. G. Grant, *Petro-chem. Eng.*, 34-40 (Nov., 1963).
8. Redmon, O. C., *Chem. Eng. Progr.*, **59**, 87 (1963).
9. Robinson, J. W., U. S. Pat. No. 2,611,490.
10. Rose, P. M., M.S. thesis, Illinois Inst. Technol., Chicago (June, 1963).
11. Sareen, S. S., M.S. thesis, Illinois Inst. Technol., Chicago (Feb., 1966).
12. Treybal, R. E., "Liquid Extraction," 2 ed., pp. 447-450, McGraw-Hill, New York (1963).
13. ———, "Mass Transfer," pp. 366-367, McGraw-Hill, New York.
14. Vinson, C. G., Jr., Ph.D. thesis, Univ. Michigan, Ann Arbor (1965).
15. Voyutskiy, S. S., K. A. Kal Yanova, R. M. Panich, and N. M. Fodiman, *Dokl. Akad. Nauk SSSR*, **91**, 1155 (1953).

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