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CONCEPTUAL DESIGN STUDY OF DEMULSIFICATION OF FUEL EMULSIONS BY A COALESCENCE PROCESS

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By

William J. Asher

April 1969

U. S. ARMY AVIATION MATERIEL LABORATORIES
FORT EUSTIS, VIRGINIA

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GOVERNMENT RESEARCH LABORATORY
ESSO RESEARCH AND ENGINEERING COMPANY
LINDEN, NEW JERSEY

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This report was prepared by Esso Research and Engineering Company under the terms of Contract DAAJ02-68-C-0011. The purpose of the effort was to investigate the feasibility of recovering military specification JP-4 fuel from emulsified JP-4 by means of a coalescence process.

The program was partially successful, in that emulsion breakdown in excess of 90 weight percent was achieved on occasion; however, the emulsifying agent tended to dissolve within the JP-4 phase, resulting in high gum level readings when the fuel was measured against MIL-T-5624G requirements. Additional effort will be required to determine if these gum levels are significant from the standpoint of turbine engine operation. Simple filtration methods removed all but a trace of the gum, but this added procedure would not be desirable from the standpoint of on-board aircraft demulsification.

Recommendations and conclusions contained in this report represent the opinion of the contractor and are partially concurred in by this Command. It is felt that further efforts of a more comprehensive nature are required before the system investigated herein can be indorsed as the optimum method for achieving emulsified fuels demulsification resulting in military specification JP-4 aviation fuel.

Project 1F162203A529
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Final Report

By

William J. Asher

Prepared by

Government Research Laboratory
Esso Research and Engineering Company
Linden, New Jersey

for

U. S. ARMY AVIATION MATERIEL LABORATORIES
FORT EUSTIS, VIRGINIA

SUMMARY

If emulsified fuels are to be used in logistical hauling, a practical method of demulsification of these fuels must be developed. This program has been carried out with this aim. In this program, the technical feasibility of continuous demulsification of safety fuel emulsions has been demonstrated. The demulsification involves the recovery of the liquid fuel from the emulsion, which can then be used for any of its normal applications; e.g., as a fuel or coolant. Demulsification involves three essential steps:

- Breakdown of emulsion.
- Coalescence of unbroken emulsion.
- Separation of unbroken emulsion from the fuel.

In this study, stainless steel wool packing or screens were used for the critical steps of breaking and coalescing. Both of these were equally effective, but the steel-wool packing is more practical. Depending on the emulsions, 79 to 92% breakdown by weight was achieved. This performance was obtained by passing 1.4 gpm of the emulsions through a small tube (1/4 inch in diameter by 1-1/2 inches long) containing the steel wool. The most important parameter determining the extent of breakdown was found to be the energy dissipated per unit volume of emulsion. The best breakdowns were obtained at the upper limit of energy dissipation of the equipment used. If this energy is increased either by operating at pressures greater than 240 psi or by using a recirculating system, the extent of emulsion breakdown should be increased.

Continuous separation of broken emulsion was demonstrated in this study using Esso WSX 7165 fuel emulsion. The separation based on the difference in specific gravities of JP-4 and unbroken emulsion functioned best with highly broken emulsion. In general, the quality of the separated JP-4 was good; however, coming directly from the separator, the JP-4 had a slightly cloudy appearance caused by about 50 ppm of small emulsion globules, which were readily removed by simple filtration. The resulting JP-4 was completely clear and met the military specifications (MIL-T-5624G) with the exception of dissolved surfactant, which conventional testing procedures classified as gum. Although the surfactant was removed by adsorption on charcoal, it is questionable whether it represents a significant debit.

Since the scale-up of packed beds is well understood, further development could lead to either practical field depot units or small, vehicle-mounted demulsification units.

FOREWORD

This report summarizes work aimed at demonstrating the feasibility of a breaker-coalescence technique of demulsifying fuel emulsions on a practical basis.

This work was initiated in October 1967 and completed October 1968. The program was administered by the U.S. Army Aviation Materiel Laboratories, Fort Eustis, Virginia, with Mr. William Nolan and Captain George Bowling acting as coordinator. We are grateful to them both for their many timely suggestions.

The work reported herein was authorized by DA Project 1F162203A529.

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INTRODUCTION

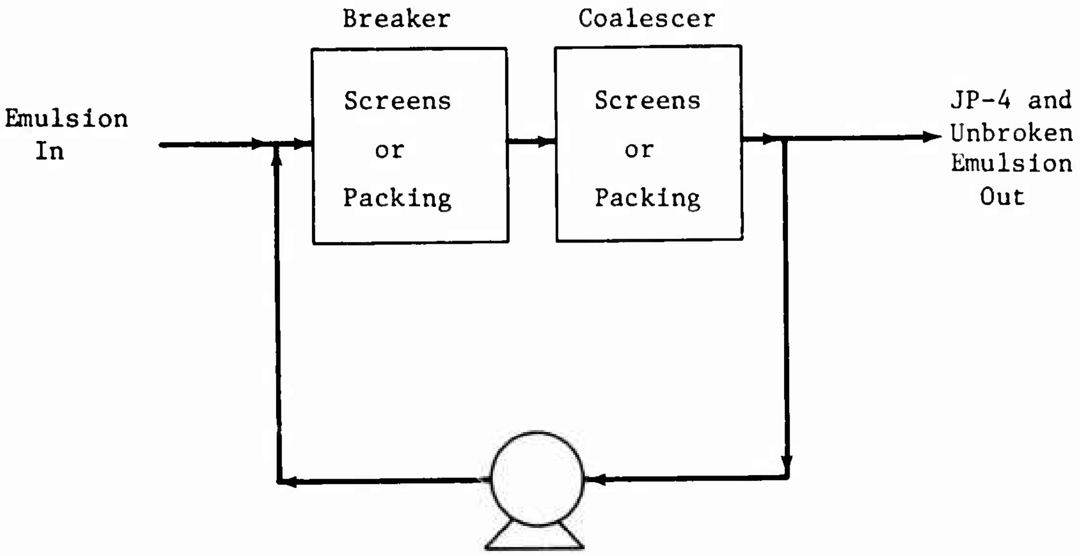
For some potential applications, demulsification of safety fuel emulsions would be desirable. Fuel transported and stored in the emulsion form will enhance safety; however, it is doubtful that all fuel-consuming devices and vehicles will be qualified to operate on emulsified safety fuels. It would thus be advantageous to be able to demulsify, i.e., break, the stored emulsified fuel and recover the liquid fuel for use in any standard device. This demulsification could be done as the fuel is removed from bulk storage and put into the tank of the device or vehicle. This approach requires a high-capacity demulsifier at the storage site. In other situations, it may be preferable to retain the emulsion safety features in the tank of the device or vehicle and to demulsify the fuel only as it is used. For this approach, a miniature demulsifier could be used with each using device.

In practical demulsification, more than a simple breakdown of the emulsion must be accomplished. The remaining unbroken emulsion must be coalesced into large globules, to facilitate separation from the fuel, and the separation effected. Preferably, this should be done with minimum weight, volume, and energy and with no addition of chemicals.

Prior to the proposal for this contract, it was noted in our work with emulsion that screens or packing could be used for the two key steps in practical demulsification-breaking and coalescing. The objective of this program was thus to demonstrate the feasibility of using this approach for a practical process for the demulsification of fuel emulsions.

EQUIPMENT FOR EXPERIMENTAL INVESTIGATION

There are two basic flow systems that can be used with screens or packing to effect emulsion breakdown and coalescence. The first is the recirculating system shown in Figure 1.



Recirculating Pump

Figure 1. Recirculating Breaker and Coalescer.

In this system, the emulsion is introduced into a loop which is circulating emulsion many times faster than the rate at which the emulsion is introduced into the loop. This loop contains the screens and/or packing used to cause the breaking and coalescing of the emulsion. Because of the very low ratio of emulsion introduction to emulsion recirculation in the loop, each entering element of emulsion is, on the average, passed through the breaking and coalescing screens or packing many times before exiting from the loop. The products of free JP-4 and unbroken emulsion are withdrawn from the recirculating stream after many passes. The second system, illustrated in Figure 2, uses only one pass through the screens or packing.

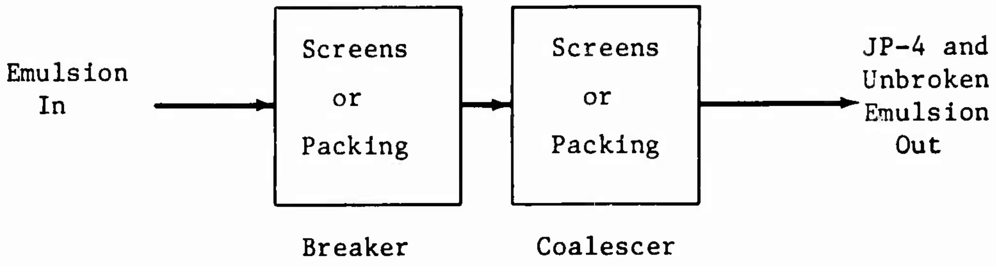


Figure 2. Once-Through Breaker and Coalescer.

In the figures, the breaking and coalescing are shown as separate and distinct zones. Of course, the functions of both breaking and coalescing could, with appropriate screens or packing, be done in one uniform zone rather than two distinct zones.

The advantages and disadvantages of the two types of systems are rather straightforward. The recirculating system utilizes the screens or packing several times and therefore should require less of them for a given level of demulsification. Also, more energy (total) can be introduced to the emulsion at the same pressure drop across the screens or packing in a recirculating system. Balancing these advantages is the disadvantage of having an additional pump for recirculation and the increased complexity of the process. A processing advantage of the once-through system is that all the entering emulsion passes through the breaker-coalescer and is thus uniformly exposed to breaking conditions; in the recirculating system, some elements of the emulsion pass through the breaker-coalescer many times while some of the emulsion passes only once, which results in exposure to different breaking conditions.

In both the recirculating and the once-through systems, the packing or screens which did the breaking and coalescing were held in cartridges which allowed variations in both the number of screens and the cross section for flow. The screens were clamped in a 1-1/2-inch-inside-diameter tube with rubber and metal washers. Metal and rubber washers of different inside diameters were used to determine the cross section for flow (see Figure 3).

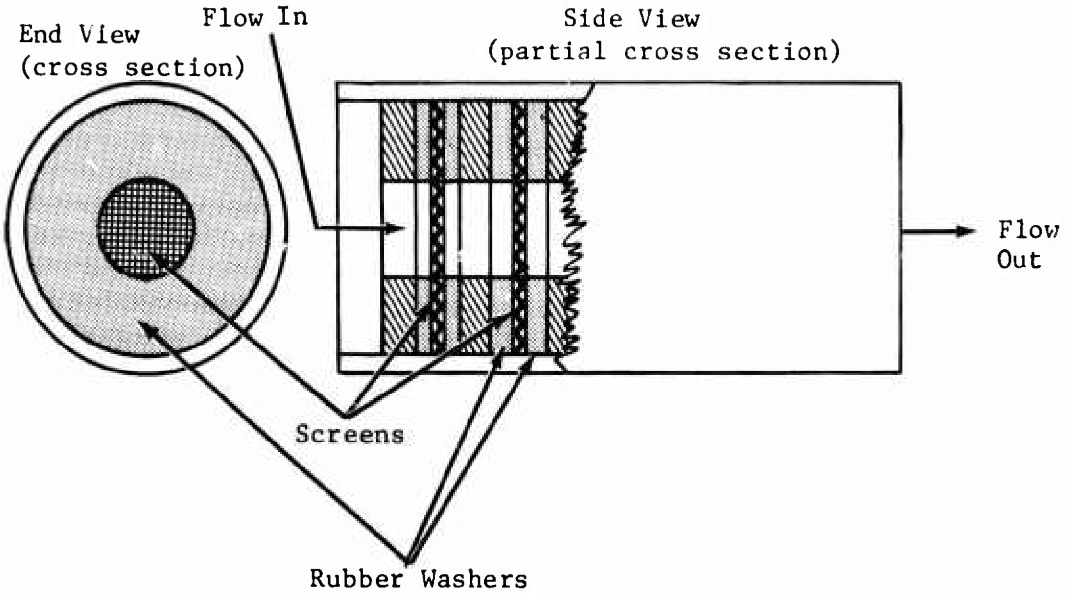


Figure 3. Cartridge for Breaker-Coalescer Screens.

When packing was used, it was simply compressed inside the tube to the desired length, and a coarse screen on the outlet end was used to retain it. To vary the cross-sectional flow area, tubes of different inside diameters were used.

When using either the packing or the screens, the emulsions were exposed to high shear rates, which are the major and perhaps the dominant mechanism by which demulsification occurs. The shear rates encountered with the use of the packing cannot be quantized; however, this is not the case when screens are used as the emulsion breaker. Even though it is extremely difficult to determine actual shear rates even when screens are used, it is possible to quantify the intensity of shear by the calculation of a pseudo-shear rate, which is what we measured and used in this program. The pseudo-shear rate is defined as the shear rate which would occur in a capillary tube having an inside diameter equal to the distance between the screen wires, with a flow through the capillary equal to the flow through each of the openings of the screen. A rigorous shear rate can be calculated only for the circular cross section of a capillary in the region not affected by end effects. Rigorous calculations of the shear rate are not possible at the end of a capillary and certainly not with the complicated geometry of crossing wires. The pseudo-shear rate, on the other hand, can be calculated and quantifies a relative shear rate which is quite useful for this study. See the Appendix for the equation relating mesh number (wires per inch), wire diameter, cross-sectional area, and pseudo-shear rate. The pseudo-shear rate varies simply with major variables as indicated below:

- Directly with flow rate.
- Inversely with the square of passage diameter.
- Inversely with the number of wires per inch.

The emulsions were fed to the systems for demulsification by a hydraulically driven, free-floating piston in a cylinder. The emulsion was charged in the cylinder on top of the piston. After the top head of the cylinder was replaced, the emulsion was forced out through a tube located at the top of the cylinder by driving the piston up with a hydraulic fluid on the bottom of the piston. (See Figure 4.)

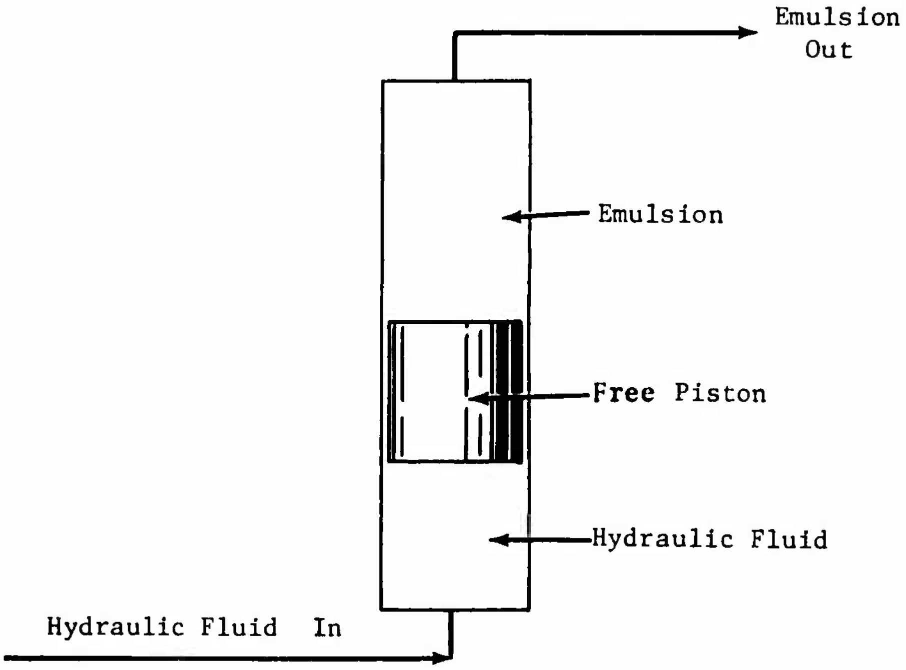


Figure 4. Emulsion Feed System.

The piston and cylinder assembly was a hydraulic accumulator with a 5-gallon capacity. No problems were encountered throughout the program with leakage past the piston seals in either direction. As all gas was vented from the system before a run, the rate of flow of hydraulic fluid into the cylinder had to equal the rate of flow of emulsion out. The hydraulic fluid flow rate was measured with rotometers and could be varied from 10 to 5300 ml/min (1.4 gpm). This technique allowed the measurement of emulsion flow rates to the unit without inserting something into the emulsion path which might either have changed the physical properties of the emulsion or have partially broken it.

The systems were evaluated in terms of the weight percent demulsification and the effectiveness of coalescence. This was done by allowing the effluent stream which contained broken JP-4 and unbroken emulsion to flow into a tray with a 20-mesh screen bottom. This screen which retained the well coalesced unbroken emulsion allowed the less well coalesced material to pass through to another tray with a 60-mesh screen bottom,where it was retained. In some cases, an 80-mesh screen was also used. The JP-4 ran through the screens into a collection pan. The materials in the pan and trays were then weighed. A continuous separator for the products was evaluated in one portion of the experimental program. This separator is described in the portion of the report evaluating its operation.

RESULTS AND DISCUSSION

Recirculating System

The first work in the demulsification studies was carried out using a recirculating system. The key to obtaining data that is useful in scaling up such an operation involves selecting a pump that will not itself break the emulsion. The absence of emulsion breaking in the pump thus allows the breaking and coalescing to occur in the screens or packing. Conditions in the screens and packing are sufficiently defined so that the critical parameters can be calculated for accurate scale-up. As high shear rates were known to partially break the emulsion, a pump with minimal shear rate was selected: a vane pump. The model selected has a rated output of 11 gpm at 575 rpm. Pump speeds from 30 to 120 rpm were used to further reduce the shear rates in the pump and to provide a range of flow conditions for the experimental program.

The initial results with the recirculating system were quite encouraging. In this test, 10 screens of 8 mesh and 0.028-inch wire diameter were used with 0.375-inch-diameter opening for flow. The emulsion used was a WSX 7165 formulation, which, although about six months old, was in good physical condition with no breakdown evident. This emulsion was introduced into the loop at a 100 ml/min flow rate. At low recirculation flows of about 0.6 gpm, no emulsion breakdown was found while yield stress of the emulsion was increased. The recirculating flow rate was increased to about 2.4 gpm, and gross breakage occurred with excellent coalescence of the unbroken emulsion. Breakdowns in excess of 90 weight percent were achieved. Coalescence was excellent, as the remaining unbroken emulsion was discharged from the exit line in globules the diameter of the exit line (1/2 inch) and 1 to 3 inches in length. However, attempts to repeat these results on the same system were unsuccessful, and examination of the breaking cartridge showed it to be plugged. In the latter runs, the emulsion breakdown dropped to about 45 to 50 weight percent. The percent of breakdown did not change when freshly prepared emulsion was used. One can infer from these results that the first screen was partially blocked during the initial tests, thereby producing shear rates higher than those expected. The emulsion was thus probably broken as it passed through the first screen and was coalesced as it passed through the remaining 9 screens. In attempts to repeat the experiments where >90 percent demulsification was achieved, variations in pressure drop over the screens as a function of time under a constant set of conditions were found and investigated. When more or smaller size screens were used, the variations in pressure drop as a function of time were accentuated. This suggested that, at some pressure drop, material was being forced back through the pump counter to flow produced by the pumping action of the pump, and the amount of backflow (pump slippage) was variable. To observe the flow in the loop, a 1-inch-long section of clear plastic tubing was inserted. Observation of the flow in the clear section thus confirmed variable flows and therefore variable backslippage through the pump. As backslippage through the pump had to occur by flow past the edges of the vanes, the vanes were spring-loaded against the periphery of the housing to keep them properly located. This largely eliminated the variation in flow and backslippage, but backslippage was still suspected. Backslippage through the pump could be a major problem because the reduced flow through the screens would lower the pseudo-shear rate at the screen and thus reduce the degree of breakdown. As higher pressure drops are required to move the emulsion in the unbroken form relative to the broken emulsion, high pressures causing backslippage which reduced the breakdown would lead to still higher pressures, increasing the backslippage to yield still lower breakdown. This "snowballing" or positive feedback effect can therefore produce a stable unit operating condition having low rather than high demulsification levels. It is thus necessary to eliminate or greatly minimize backslippage through the pump.

Backslippage through the pump was tested in the unit using both WSX 7165 emulsion and JP-4; the use of JP-4 represented a severe test for backslippage. Although a safety fuel emulsion has Newtonian equivalent viscosity infinitely high at shear stresses below the yield stress, previous rheological studies* have shown the effective flow properties to be near those for JP-4 under extremely high shear stress conditions. The tests for backslippage were run at various pump speed settings and at backpressures of 0 and 40 psi. Backslippages were shown to be quite large at the 40 psi backpressure. (See Figure 5.)

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* Nixon, J., et al., "Investigation and Analysis of Aircraft Fuel Emulsions," Esso Research and Engineering Company; USAAVLABS Technical Report 67-62, U.S. Army Aviation Materiel Laboratories, Fort Eustis, Virginia, November 1967, AD 827051.

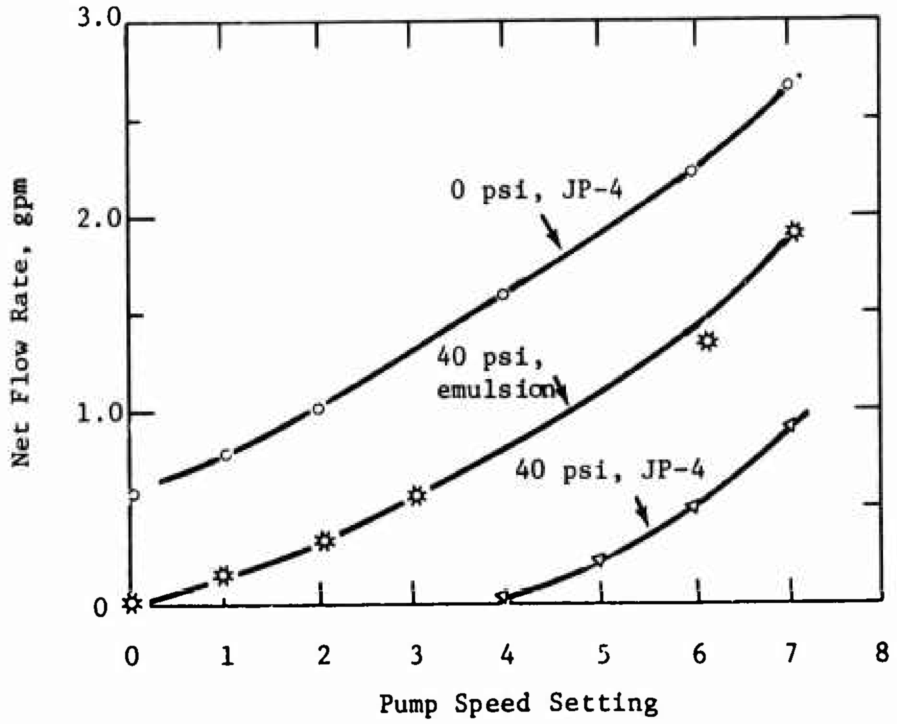

Figure 5. Flow Data Showing Effect of Pump Slippage.

At pump settings that should have produced flows of 1.6 gpm, a 40-psi backpressure reduced the net JP-4 flow to zero. The backslippage with the emulsion was almost as bad, showing that under these conditions the flow behavior of the emulsion was quite similar to that of JP-4 rather than that of a highly viscous liquid.

To determine if dimensions outside the manufacturer's tolerances were responsible for the large backslippage, the pump was disassembled and all dimensions were measured with micrometers. All were found to be within the manufacturer's tolerances. In addition, pump backslippage data using gasoline was obtained from the pump manufacturer. This data showed essentially the same slippage that was obtained in this laboratory using the JP-4. On this basis, it was concluded that the pump was performing within the specification. Although this pump will deliver very viscous fluid well at pressures of 40 psi or higher, backslippage is a major problem with the emulsions because, under the conditions in the pump, they behave like JP-4, a low-viscosity fluid.

Successful operation of the recycle system using this pump is therefore apparently dependent on a sensitive balance. The breakage in the first screen must be high enough that the pressure drop in the subsequent screens is low enough to prevent substantial backslippage in the pump. Rather than await arrival and testing of other low shear pumps which might avoid this problem or use a high shear pump, which would make data interpretation questionable, the program was shifted to the once-through system. The once-through system, of course, requires no recycle pump and has the major advantage of simplicity.

Once-Through Operation With Screens

When the program was shifted to the once-through system, finer screens (higher mesh) had to be used to attain the same pseudo-shear rates as with the recirculating system. As will be recalled, the pseudo-shear rate varies directly with flow rate and inversely with distance between screen wires. The emulsion feed system had a maximum delivery rate of only 960 ml/minute (.25 gpm), compared to the recirculating system maximum rate of 2.4 gpm. To compensate for the lower flow rate, the distance between screen wires had to be reduced (increased mesh number).

Particulate contamination in the WSX 7165 emulsion, which was produced prior to the special procedure to minimize contamination, caused severe operating problems. Screens of 120 mesh plugged in seconds when used at high pseudo-shear rates. The level particulate contamination was moderate, as only 7 weight ppm were retained on a 400-mesh screen. Analysis indicated that iron was the major component. As the rapid plugging had to be eliminated, attempts were made to remove the particulate contamination by filtration. An unusual phenomenon plagued all of the filtration work. This phenomenon was the plugging of screens used in breaking the emulsion after the emulsion had been passed through finer screens or filters upstream, which should have removed any particles capable of plugging the breaking screen. This phenomenon is exemplified by emulsion which had passed through a 400-mesh screen plugging a 250-mesh screen. The emulsion flowed through a .8125-inch-diameter cross section of 400-mesh screen at 350 ml/minute. As the pseudo-shear rate produced was only 13,000 sec $^{-1}$, the quantity broken was small, about 10 weight percent. This screen should have removed contaminant particles of 30.5 microns and larger. The emulsion then flowed through a 250-mesh screen, which should not have retained particles smaller than 61 microns. The diameter of the cross section for flow in this second screen was .25 inch, and a pseudo-shear rate of 86,000 sec $^{-1}$ was produced which gave an initial breakdown of 46 weight percent. In an average time of 30 minutes, the system plugged; and it was the second, coarser screen which was blocked. As the extent of demulsification increased, the rate of pressure drop increase accelerated. The system rapidly became blocked at the end of a run when the breakdown was in the range of 80 to 85 weight percent. A mechanism consistent with this performance is as follows. A substantial portion of the particulate contamination could have been particles smaller than 30 microns which would pass through a 400-mesh screen. Particles in emulsion generally tend to be held by the surfactant between the continuous and discontinuous phases. In the case of the safety fuel emulsion, the particles tend to be isolated from each other because of the yield stress, thus minimizing coagulation of the particles. However, when the emulsion passes through a screen at high shear rates and is broken, the particulate particles are released and are allowed to come together and agglomeration results. The agglomerated particles are then large enough to block the second, coarser screen. If this mechanism is correct, it means that the filtration of emulsion under conditions that will not cause them to break may very well require finer filters than would be required to filter the same contaminates from a liquid where agglomeration can occur.

Although filtration did not eliminate the plugging of the screens used for breaking, it did greatly alleviate the problem. This is indicated by the increase in the time to plug a 250-mesh screen having a .25-inch-diameter passage with 350 ml/min emulsion flow. The time to plug was extended from an average of 20 seconds to 30 minutes by inserting a 400-mesh filter screen. Filtration was thus used so that breakdown data could be obtained with screens of smaller holes than the 20-mesh screen used in the recirculating system program. In addition, emulsion flow rate was increased from 0.25 to 1.4 gpm to allow higher shear rates with screens of moderate mesh size. Since breakdown in the filters is small relative to that achieved in the screen, the extent of breakdown measured is a characteristic of the screen under test. The need for higher maximum flow rates and the need to remove reasonably small particles with minimum emulsion breakdown are stringent filter requirements. Both higher flow rates and finer screens or filters, of course, increased emulsion breakdown. Increasing the area for filtration reduced the problem, but there are practical limits. A compromise filtration system was thus selected which consisted of three filters in parallel to maximize filter area. Each filter was fitted with a filtering element 3/4 inch in diameter and 1-1/2 inches long, with filtering surface on the walls and one end of the cylinder. These bronze elements were rated from 50 to 125 microns. The tests of breakdown versus flow rate are shown in Figure 6.

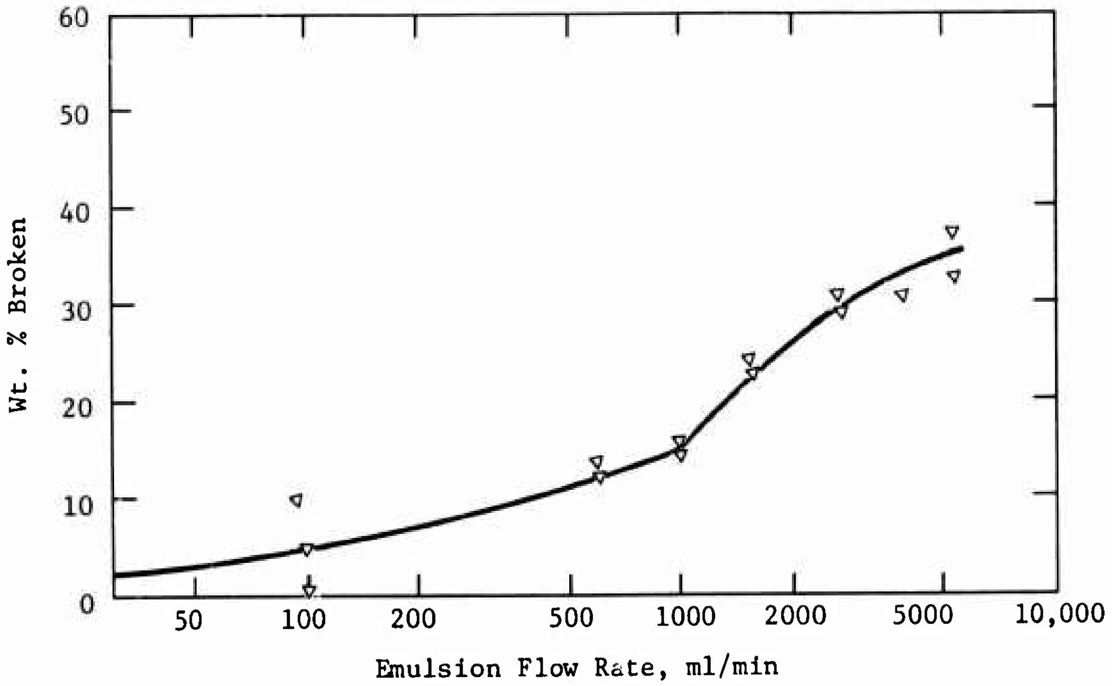

Figure 6. Effect of Filtration Rate on Emulsion Breakdown.

This filtration system did not distort the breakdown data. No difference in breakdown was measured in later tests with and without the filter upstream from a cartridge yielding 85 weight percent breakdown at the 5300 ml/min rate.

The filters described also allowed data to be obtained with fine screens. However, with screens finer than 60 mesh, it was very difficult to obtain data that was not influenced by partial blockage of the screen. In many cases, the pressure drop and percent breakage would rise from the time that flow was started. An example of the progression is seen in an experiment using a 250-mesh screen with 180 ml/min of flow going through a .25-inch-diameter passage. The initial pressure drop was 10 psi, and there was only nominal breakage. After 7 minutes, the pressure drop had increased to 109 psi and the breakdown was 85.8 weight percent. A further complication was associated with the particulate contamination's not being uniformly dispersed in the batch of emulsion. At 180 ml/min flow through a .25-inch-diameter passage with a 250-mesh screen, the same emulsion sample required 30 minutes, 1 minute and 15 seconds, and in one case 15 seconds to complete blocking of the screen. The unifying characteristic when using the fine screens was that 80 to 85 weight percent breakage was achieved when the system stopped flow at 110-psi pressure; this was independent of screen mesh or the initial diameter of the screen exposed for flow.

Work with the coarser screens (60 mesh) was not interrupted with problems caused by blockage. When using a 2250 ml/min flow, which corresponds to a pseudo-shear rate of 135,000 sec^{-1} through a .25-inch-diameter passage, 65 weight percent breakage was achieved. Increasing the number of 60-mesh screens from 1 to 10, which increased the extent of shear without any appreciable change in shear rate, increased the percentage of breakage to 81.5 weight percent. This shows the benefit of increasing the extent of shear. Figure 7 shows the increasing emulsion breakdown as the emulsion flow rate is increased when operating with the 10 screens in series. Since the shear rate increases with increasing flow rate, this data shows the expected increase in extent of breakdown with increasing shear rates.

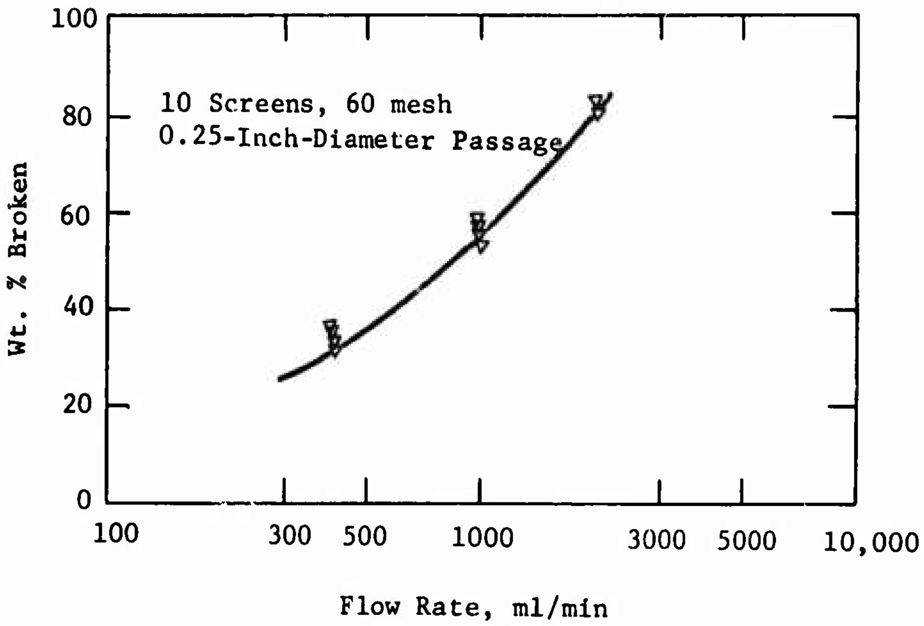

Figure 7. Effect of Having Screens in Series on Emulsion Breakdown.

The fabrication of large devices containing many screens is both complex and costly. Approximating the action of many screens in series with a packing is thus a preferable engineering route. The program was therefore shifted to the study of the use of packings as breaking devices for emulsions.

Once-Through Operation With Packing

The experimental program to investigate the use of packing as the breaker in the demulsifier unit was designed to determine the most important parameters as well as to explore the effectiveness of packing for breaking and coalescing the safety fuel emulsion. The packing investigated was made of stainless steel, selected to eliminate corrosion from the atmosphere as well as the safety fuel emulsions. The packing was a fine grade stainless-steel wool that was compacted to a cartridge with a 0.25-inch inside diameter. In the program, the effect of varying degrees of packing compactness was investigated by using a fixed quantity of the steel wool, 1.5 grams, compressed to various lengths between 1 and 2-3/4 inches. The emulsion flow rate was then varied with each packing length, and the breaking and coalescing performance of the packing were measured.

The data from this experimental program can be analyzed in several ways. One analysis was done using packing length as a parameter by plotting the percentage of breakdown against superficial linear velocity in the packing zone. The superficial linear velocity is defined as the fluid velocity under plug flow conditions through an empty tube having the same inside diameter as the cartridge holding the packing. The plot in Figure 8 shows that increasing either the superficial velocity or the degree of compactness increased the extent of demulsification.

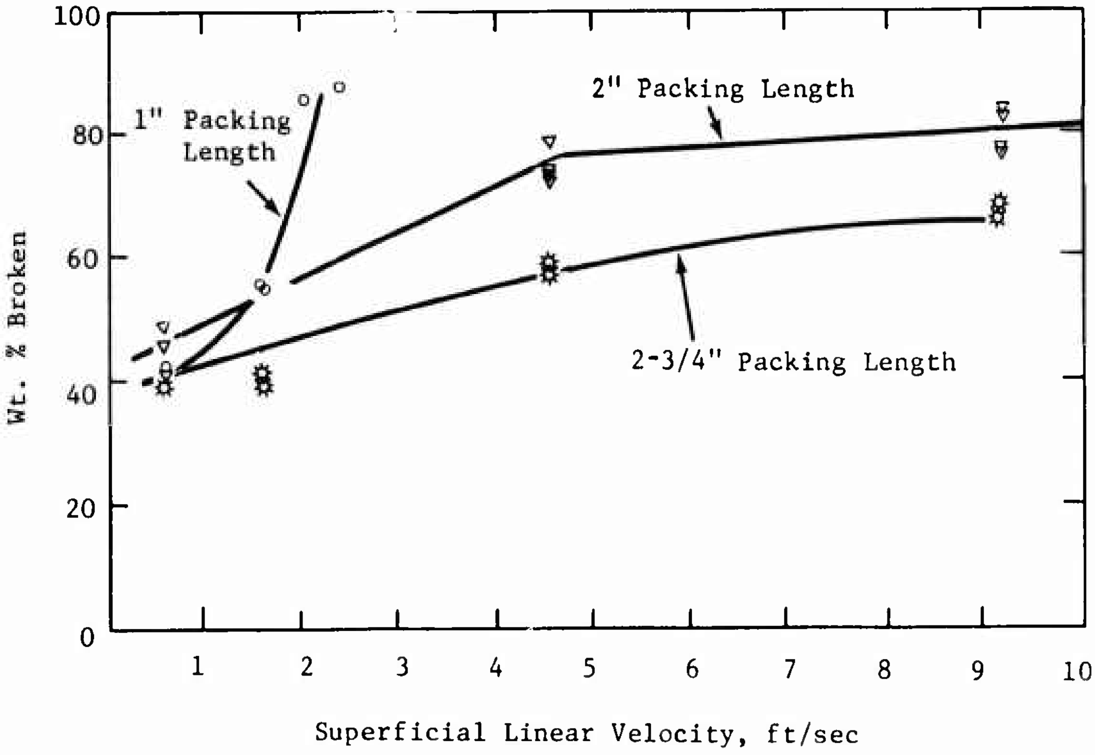

Figure 8. Effect of Superficial Velocity on Emulsion Breakdown.

For a given superficial velocity, the more compact the packings, the higher the pressure required to force the emulsion through the packing.

Another way to evaluate the demulsification involves using the pressure drop required to produce the observed degree of demulsification. When all of the data, independent of linear velocity or packing length, were plotted with percent breakage versus pressure drop, a single line could be drawn through all the data. (See Figure 9.)

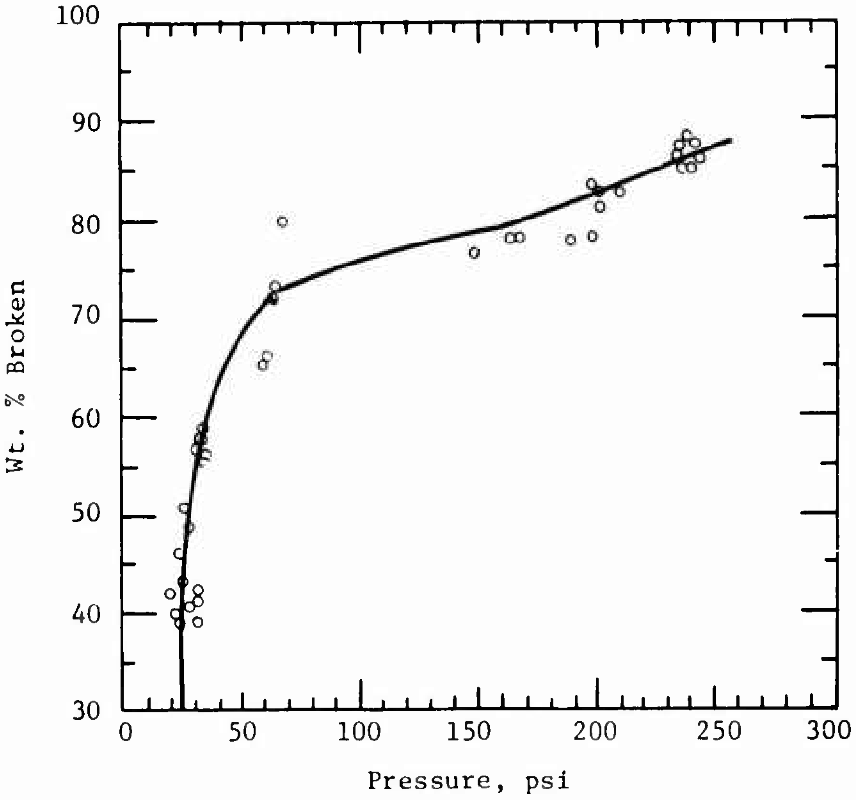

Figure 9. Effect of Pressure Drop on Emulsion Breakdown.

Thus, the extent of demulsification was found to be independent of either superficial velocity or packing length alone and could be expressed as a function pressure drop only. Since at constant pressure drop the energy dissipated per unit volume is constant, this finding means that the energy dissipated per unit volume of emulsified fuel passing through the packing is the most important parameter affecting the extent of demulsification.

However, the energy dissipation or pressure drop does not define the percentage of breakdown independent of the device used. This is exemplified by the data obtained in runs made using the same 0.25-inch-inside-diameter tube packed with tetrafluoroethylene filaments rather than the stainless-steel wool. With the tetrafluoroethylene-packed tube, the percentage of breakdown is considerably lower than that obtained with the steel-wool-packed tube at the same pressure. (See Figure 10.)

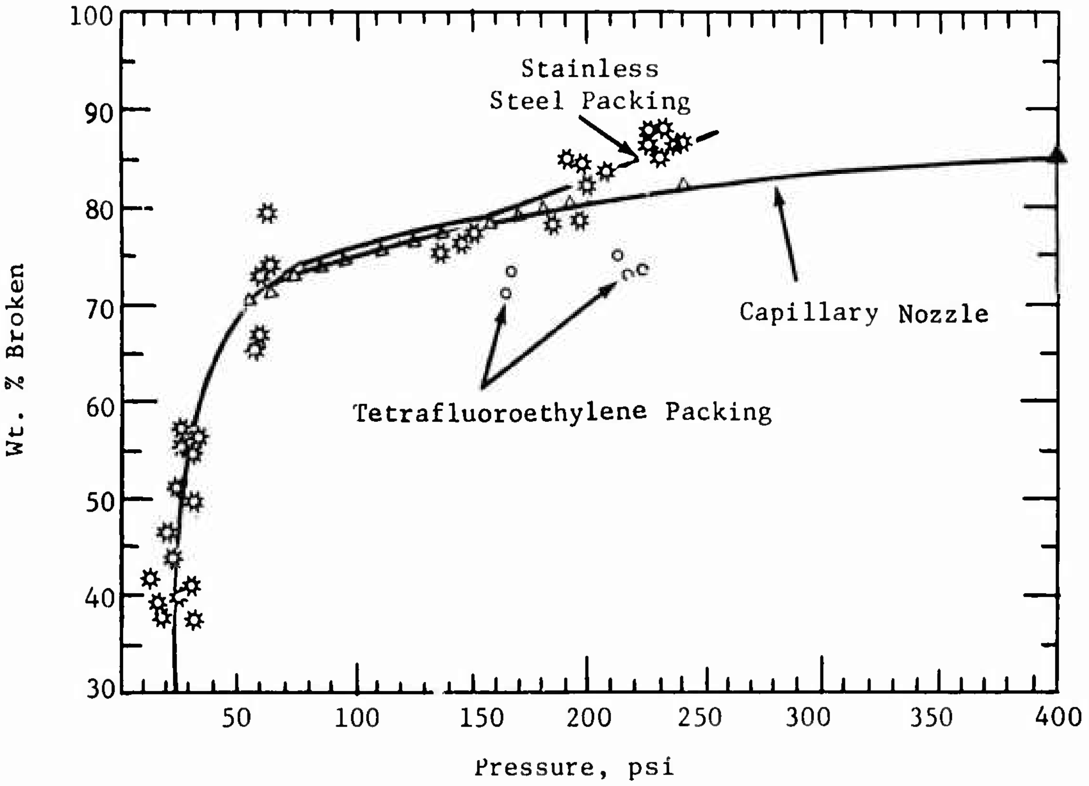

Figure 10. Comparison of the Efficiency of Various Demulsification Methods.

The performance difference between tetrafluoroethylene and the steel-wool packings is thought to be attributable to the different wetting characteristics of the emulsion with respect to these two materials. The tetrafluoroethylene is preferentially wetted by the discontinuous phase (i.e., the JP-4), while the steel wool is preferentially wetted by the continuous phase. The advantage in using a packing wetted by the continuous phase is thought to be as follows. The unbroken portion of the emulsion will tend to adhere to surfaces wetted by the continuous phase; thus, the emulsion tends to collect on the steel wires where the local shear rate is at a maximum. In contrast, the local shear rate goes to zero midway between two strands. Thus, the unbroken portion of the emulsion is exposed to the maximum shear rates, which tend to produce maximum breakdown for a given set of flow conditions. However, when the packing is wetted by the discontinuous JP-4 phase, as is the case with tetrafluoroethylene, the unbroken portion of the emulsion does not adhere to the strands, and it is therefore exposed to shear rates which are less than the maximum. This, therefore, tends to produce something less than maximum breakdown for a given set of flow conditions.

It is also interesting to compare the extent of demulsification using the steel-wool packing with that obtained by forcing the emulsion through an orifice such as that being studied in the rheological portion of another contract.* In that study, a capillary tube with an inside diameter of 0.01165 inch and a length of 0.033 inch was used to simulate one of the holes in an engine nozzle. A plot of some of the data obtained on WSX 7165 in that program is also shown in Figure 10, where the percentage of breakdown is plotted against pressure drop across the capillary. While the nozzle approach gave greater demulsification than that obtained using the breaker-coalescer containing tetrafluoroethylene packing at equivalent pressure drops, the nozzle was not as good as the percentage of breakdown achieved with stainless-steel packing in the breaker-coalescer. At the 85 percent demulsification level, the breaker-coalescer with steel wool required almost a 200-psi lower pressure drop relative to the nozzle approach to demulsification.

Packing Scale-up

An advantage in using packing for demulsification is that the state-of-the-art engineering will allow straightforward design of equipment scaled up to any flow rate. In a packed bed, the flow dynamics and, therefore, the results of the flow are identical, independent of the design flow rate, when the proper parameters are held constant. These parameters are the depth in the direction of flow and the flow rate per unit of cross-sectional area perpendicular to flow. Thus, in scaling up data obtained at 1.4 gpm in a 0.25-inch-inside-diameter tube 1.5 inches long, the length is kept the same and the diameter of the tube is sized, keeping the flow rate per unit area the same. Using this procedure for a 400-gpm unit, a disk 4.25 inches in diameter with the flow going through a 1.5-inch depth is obtained. The pressure drop over this device would be the same, 240 psi, measured with a small 1.4-gpm unit. In addition, the percentage of breakdown would be the same, 86 percent. Since the pressure drop would be the same for any size unit, the power required per unit flow rate can be expressed as a constant, 0.128 hp/gpm in this case. The powers required for the 1.4-gpm unit and 400-gpm units are 0.18 and 51 hp, respectively.

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*Nixon, J., et al., "Optimization of Non-Aqueous Fuel Emulsions," Esso Research and Engineering Company; USAAVLABS Technical Report 69-26, U. S. Army Aviation Materiel Laboratories, Fort Eustis, Virginia.

While packed bed technology will allow good estimates for any size unit, two points of caution are necessary in using the data from this limited program as a basis for scale-up. First, it was necessary to use a very small diameter (.25 inch) tube to obtain the required flow rate per unit area. With such small-diameter tubing, it is possible that wall effects were significant. A wall effect is, of course, caused by somewhat different flow characteristics in the packing adjacent to the wall compared to flow characteristics in the bulk of the packing. Without additional experiments to quantify the wall effect, inaccurate scale-up could result. Secondly, a phenomenon was observed when runs were made using packing, which indicates that a different coarseness of steel wool may be required for practical long-term operation. The phenomenon was a slow increase in pressure drop with time at a constant flow rate. This increase could have been caused by any of three mechanisms:

1. Gross compacting of the bed of steel wool.

2. Fibers of the steel wool aligning to cause higher pressure drop.

3. Accumulation of particulate contamination from the emulsion.

Gross compacting of the bed can be eliminated, since measurements of the bed length showed that no significant change occurred. The alignment of the fibers is also a possible cause of the increased pressure drop observed. Larger diameter fibers might be required to limit the alignment. Of course, the observed increased pressure drop could have been caused simply by an accumulation of particulate matter in the stainless-steel wool. However, since this phenomenon was observed with all four of the emulsions investigated, one of which was essentially free of particulate contamination, blocking of the packing by contaminants was probably not the cause of the observed phenomenon. In addition, no visible particulate contamination was found in the steel-wool beds when they were removed.

Even though fixed-bed technology will allow scale-up to any size demulsifier, the present data is adequate only for good estimates for scale-up. Further studies would be required for accurate scale-up with confidence and to prove the long-term operability of any particular packing.

Continuous Separation

In any demulsification process, in addition to breaking and coalescing the emulsion, the liquid phase (JP-4) must be separated from the unbroken emulsion; and for practical processing, the separation must be done continuously. The continuous separator selected for this study separated the two phases, JP-4 and unbroken emulsion, on the basis of their difference in specific gravity. The separator consisted of a simple piece of glass pipe mounted with the axis vertical, which allowed the denser unbroken emulsion to move to the bottom while the JP-4 moved to the top. The separator is shown in Figure 11.

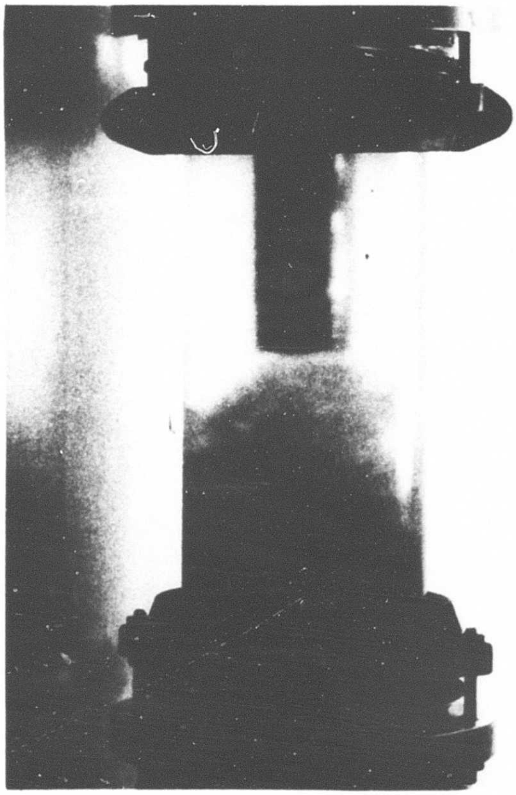

Figure 11. Glass Pipe Separator.

For its evaluation, the separator was mounted downstream from the breaker-coalescer unit. The breaker-coalescer was made up of 1.5 grams of fine stainless-steel wool packed in a 1.5-inch length of a 0.25-inch-inside-diameter tube. The safety fuel emulsion (WSX 7165) was pushed through the breaker-coalescer at 0.7 gpm, where about 80 to 85 percent demulsification was achieved. The broken JP-4 and coalesced unbroken emulsion went from the breaker-coalescer to the separator via a 1-inch pipe, which ran through the top plate of the separator and extended down about 5 inches into the 12-inch separator. The inside diameter of the separator was 4 inches. The large diameter relative to flow rate of broken JP-4 was used to minimize the upward JP-4 velocity, thereby not entraining substantial quantities of unbroken and uncoalesced emulsion in the leaving JP-4.

The separator is shown in operation in Figure 12. Also shown in the figure are the JP-4 and unbroken emulsion being continuously withdrawn from the top and bottom of the separator, respectively.

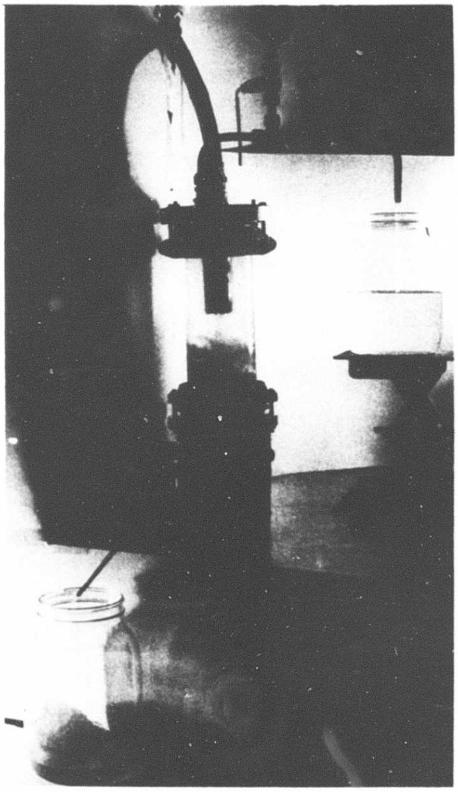

Figure 12. Separator in Operation.

The operation of the separator during these feasibility tests showed both strong and weak performance areas of this first device. The separation of the JP-4 broken from the emulsion in the separator was performed quite well. The upward velocity of JP-4 leaving was low enough to prevent any entrainment of visible unbroken emulsion of globules. The lack of any entrainment throughout the testing suggests that higher velocities, i.e., smaller diameters, would probably work just as well. However, the JP-4 leaving the separator did contain residual emulsifier. Although the emulsion droplets are too small to be seen with the naked eye, they give the JP-4 a cloudy appearance. The degree of cloudiness does not change with height in the separator, which shows that these droplets are not settling out and that a separator less than 12 inches in height would be just as effective. These cloud-forming droplets in fact settle quite slowly, with no visible settling in 24 hours in a collection vessel. However, they are easily and completely removed by filtration. Passing the JP-4 from the separator through either Whatmann No. 1 or No. 42 filter paper completely removes the approximately 50 weight ppm of material responsible for the cloud and yields completely clear JP-4.

The separation and removal of unbroken, coalesced emulsion is the more difficult step and is thus more of a challenge. The unbroken emulsion left the bottom of the separator in a stream containing from over 50 percent to less than 20 percent broken JP-4, depending on operating conditions. A straightforward solution to this undesired loss of JP-4 would be the addition of a second separator stage. The second-stage separator should work well, as the JP-4 and unbroken emulsion remained distinct phases and the JP-4 poured away from the emulsion quite readily. The principal reason that JP-4 comes out with the emulsion is the development of channels through the emulsion that allow the JP-4 to flow out the bottom. The unbroken emulsion retains a yield stress and tends to stay in place. Operation at a pressure of about 5 psig, rather than just enough above atmosphere to obtain flow, minimized the problem. The pressure apparently provides the force to overcome the yield stress. Under these conditions, the emulsion stream contained less than 20 percent JP-4. Providing surfaces that the emulsion will not adhere to, i.e., tetrafluoroethylene, polyethylene, or polypropylene, in the emulsion collection and discharge section of the separator should further minimize the tendency of the emulsion to remain in the separator. Perhaps the most significant observation was that the separator operated better when higher percentage breakdowns were achieved in the breaker-coalescer. This suggests that further research to increase the percentage of breakdown could lead to a better separator operation. This, in turn, would minimize the size of a second-stage separator or eliminate the need for a second stage altogether.

Quality of Demulsified JP-4

The quality of the demulsified JP-4 is obviously important to assure proper operation of the using device. As can be seen in Table I, the demulsified fuel obtained from WSX 7165 meets most of the military specifications for JP-4 (MIL-T-56G4G).

TABLE I. COMPARISON OF BROKEN EMULSION TO JP-4 SPECIFICATIONS

Requirement	MIL-T-5624G Spec. Grade JP-4	JP-4 Broken From WSX 7165 Emulsion
Gravity, min	45.0	50.3
max	57.0	
Existent gum, mg/100 ml	7	104
Total potential residue, 16-hour aging, mg/100 ml	14	84
Sulfur total, weight max	0.4%	0.0031%
Mercaptan sulfur weight max	0.001%	0.00031%
Reid vapor pressure, 100°F		
gm/cm^2 min	2.0	1.20
gm/cm^2 max	3.0	
Freezing point max	-72°F	<-80°F
Aniline-gravity product, min	5,250	6,036
Aromatics, vol percent max	25.0	21.7
Copper strip corrosion ASTM classification, max	No. 1	1
Thermal stability		
Change in pressure drop in 5 hours, in. of Hg, max	3.0	0.7
Preheater deposit less than	3	4 rating on 4 out of 13 sections

The Reid vapor test did not meet specifications due to extraneous handling rather than the processing. In this test, the 1.2 gm/cm^2 rather than the minimum 2.0 gm/cm^2 indicates a loss of "light ends" or low boiling materials from the JP-4. Since it is impossible to lose material in the closed breaker-coalescer and separator, this loss must have been due to excessive exposure to air either as the emulsion was being made or after it was demulsified.

A characteristic deviation of the demulsified JP-4 from the specification is the high apparent gum levels of the demulsified fuel. The high apparent gum levels have been deduced to be the result of small quantities of emulsifiers dissolved in the demulsified JP-4. As contrasted to suspended materials, the level of dissolved material would be the same with any demulsification process. Thus, dissolved emulsifier leading to high apparent gum levels would be expected to be a characteristic of demulsified fuels in which the emulsifier has a high enough molecular weight to be classified as gum. How serious these high apparent gum levels are, which are really emulsifier or surfactant levels, is open to question.

It was deduced that the high apparent gum levels were from dissolved material rather than suspended material. As will be recalled, the demulsified fuel leaving the separator was cloudy. The readily seen cloud was found to be caused by only 50 weight ppm of suspended droplets, which were easily removed by filtration. The resulting product was completely clear. Since the 104 mg/100 ml of existent gum would be equivalent to 1340 weight ppm compared to the readily visible 50 weight ppm, it is extremely unlikely that this quantity of material was suspended in the demulsified JP-4. It is more likely that the material responsible for the high existent gum was in solution. Further, consistent with this is the fact that the emulsifiers used in making emulsions must be at least somewhat soluble in the JP-4 as well as the continuous phase. The emulsifiers are typically high enough in molecular weight to be classified as gum by the normal test procedure.

Since solutions cannot be separated by mechanical means, a simple processing scheme was used to reduce the gum level. Adsorption of the high-molecular-weight emulsifier from the lower molecular weight fuel was accomplished with a charcoal which has selectivity for higher molecular weight compounds. In this experiment, 200 ml of demulsified fuel was passed through 30 grams of 20-by-40-mesh carbon. The existent gum was reduced from 104 to 7.6 mg/100 ml, very near the 7.0 mg/100 ml specification. The potential gum was reduced more than threefold, also bringing it much nearer to specification. (See Table II.)

TABLE II. GUM REDUCTION BY ADSORPTION

	JP-4 Broken From WSX 7165 Emulsion	After Charcoal Adsorption
Existent gum, mg/100 ml	104	7.6
Total potential residue 16-hour aging, mg/100 ml	84	24.4

The effect of these emulsifiers, which are classified as gum by standard testing procedure, on fuel system components should be investigated. There may be many instances where the presence of the small quantity of emulsifiers may not be of any consequence. Additionally since the emulsifier also acts as a surfactant, it may in some instances be beneficial in functioning to keep the systems clean. In light of the increased weight and complexity of chemical processing required to remove the dissolved emulsifier, this study is warranted before processing of the demulsified fuel is undertaken.

Demulsification of Other Emulsions

The general applicability of any demulsification process is important, as no one safety fuel emulsion has been selected for use. In addition to Esso fuel emulsion WSX 7165, which was the base emulsion used in this program, three Government-supplied emulsions were used in evaluating the critical breaking and coalescing steps of this demulsification process. The breaker-coalescer used was the standard one used on the WSX 7165 fuel emulsion. The Government-supplied emulsions shall be referred to herein as Emulsion A, Emulsion B, and Emulsion C.

Two of these emulsions, Emulsion A and Emulsion B, broke more completely than WSX 7156, particularly at low pressures. See Figures 13 and 14.

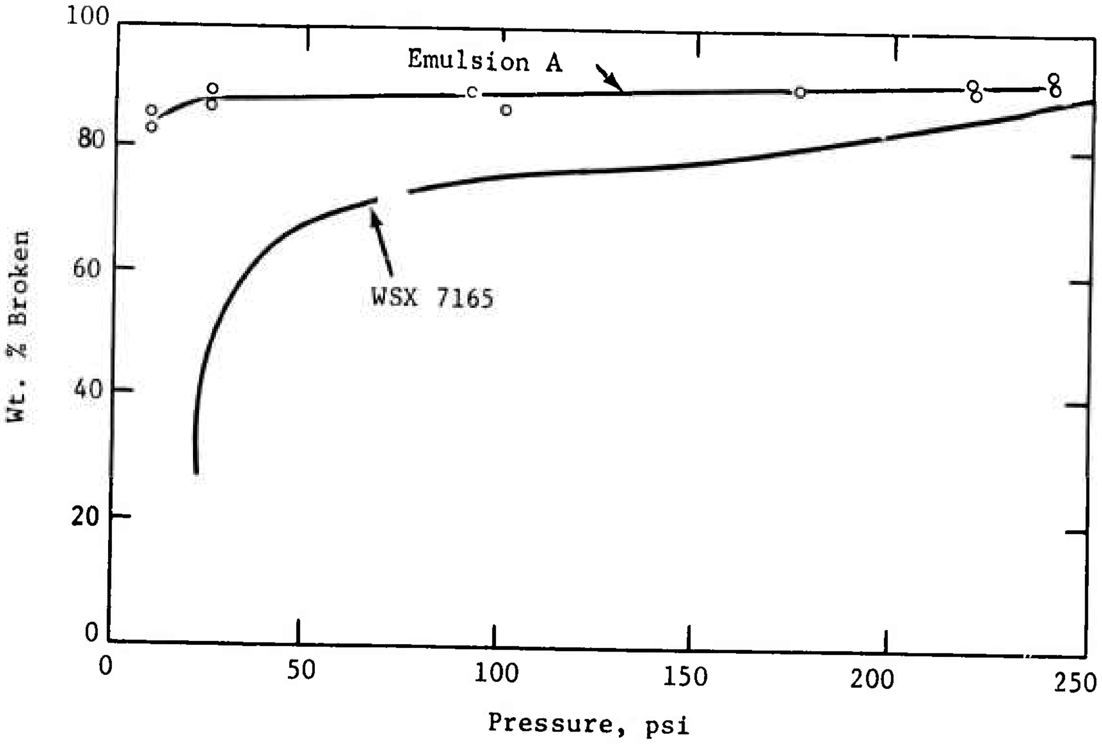

Figure 13. Effect of Pressure on Demulsification of Emulsion A.

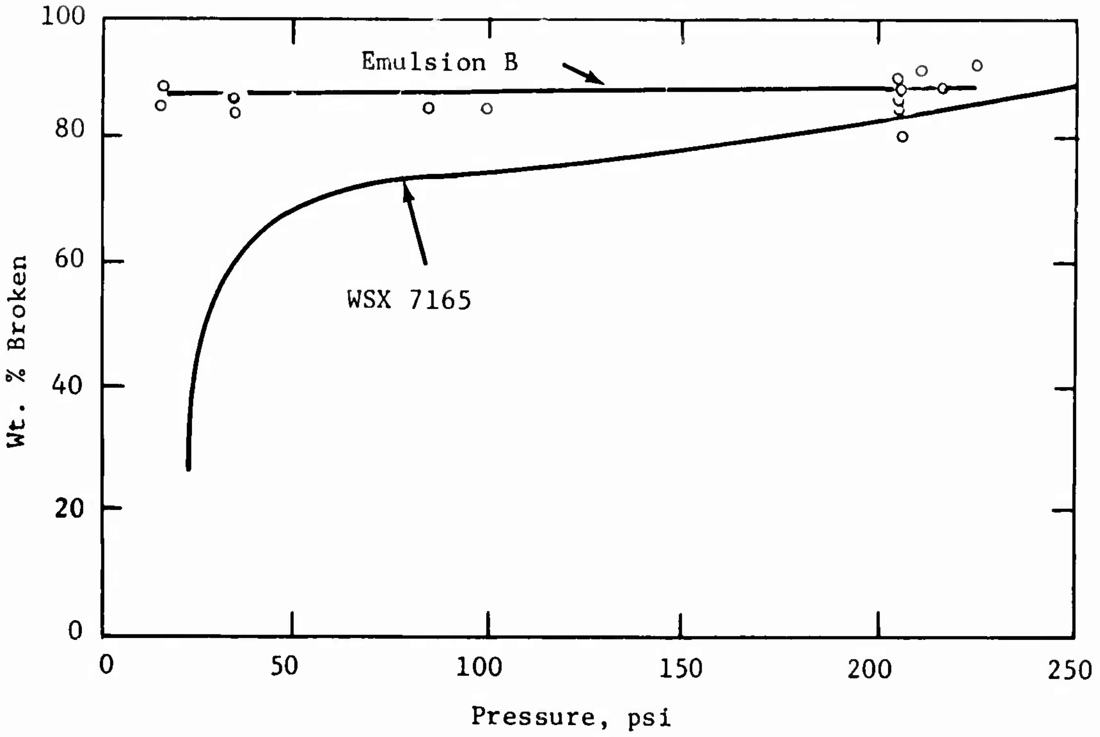

Figure 14. Effect of Pressure on Demulsification of Emulsion B.

Using the Esso demulsification scheme, a pressure of only 240 psi was required to obtain approximately 90 weight percent demulsification of Emulsion B.

Although Emulsion A and Emulsion B proved easier to break than WSX 7165, they did not coalesce as well. When operating with WSX 7165, the unbroken emulsion was coalesced well enough after leaving the breaker-coalescer so that more than 90 weight percent of it was retained on a 20-mesh screen. When operating with either Emulsion A or Emulsion B, less than 10% of the unbroken emulsion was sufficiently coalesced to be retained by the 20-mesh screen. The less effective coalescence indicates that the separator would have to be larger to effectively separate the finer emulsion globules.

Emulsion C was even more difficult to break than the standard WSX 7165. As shown in Figure 15, the breakdown for this emulsion was less at all pressure drops investigated. The coalescence of this emulsion was at least as good as that with WSX 7165, as very little of the unbroken emulsion passed through the 20-mesh screen.

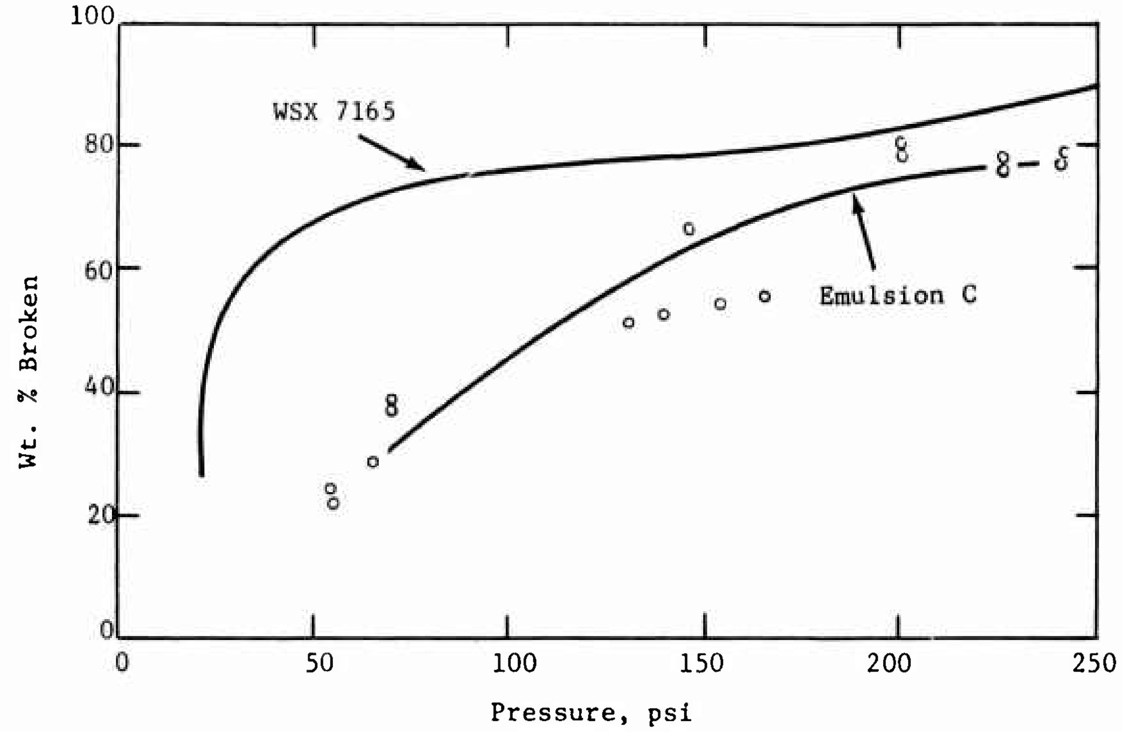

Figure 15. Effect of Pressure on Demulsification of Emulsion C.

It is interesting, although perhaps a coincidence, that the breakdown achieved at the highest pressure drop used (240 psi) decreased as the yield stress of the emulsion under study increased. (See Table III.)

TABLE III. EMULSION BREAKDOWN ACHIEVED

Emulsion	Yield Stress (dynes/cm^2)	Breakdown (wt.%)
A	690	92
B	725	89
Esso WSX 7165	1420	86
C	2185	79

Thus, the general applicability of the packing demulsification technique has been established by effective breaking of all four of the safety fuel emulsions selected by the U.S. Army Aviation Materiel Laboratories.

CONCLUSIONS

1. The technical feasibility of continuous demulsification, including all the critical steps - breaking, coalescing, and separating - has been demonstrated.

2. The general applicability of the flow-through packing process has been established by breaking and coalescing all four types of emulsions under consideration.

3. While screens have been shown to be effective, they offer no advantages over packing and would be both considerably more expensive and complex to fabricate.

4. Although the recirculating system initially gave very encouraging results, equipment problems prevented a direct comparison with the once-through system which was used for most of the program.

5. The most important parameter determining emulsion breakdown is the energy dissipated per unit volume of emulsion.

6. The energy dissipated per unit volume can also be expressed as power required per unit flow rate. In this program, 0.128 hp/gpm was the largest value used, which is quite small.

7. The extent of demulsification obtained using a once-through system, which was equipment-limited to a maximum pressure of 240 psi (equivalent to 0.128 hp/gpm), was 79 to 92 weight percent, depending on the emulsion used.

8. Both energy and the pressure required with this process at equivalent breakdowns are only about one-fourth that of the earlier reported processes.

9. Increased demulsification should be obtained by increasing the energy dissipation through either increased pressure or the use of the recirculating system.

10. The demulsified fuel from WSX 7165 emulsion meets military specifications with the exception of dissolved emulsifiers, which are classified as gum by conventional test procedures.

11. Although the emulsifiers in the demulsified fuel from WSX 7165 emulsion can be removed by adsorption, it is questionable whether their removal is really necessary.

12. Scale-up of the breaker-coalescer to any flow rate is straightforward, once the basic data is obtained.

13. A unit capable of breaking and coalescing 400 gallons per minute of emulsion would be quite small: 4.25 inches in diameter by 1.5 inches long.

14. Further development could lead to practical field depot units or to very small vehicle-mounted demulsification units.

RECOMMENDATIONS

1. A study should be made of the use of a recirculating system and the use of a once-through system at pressures higher (> 240 psi) than those investigated in this exploratory study. Only after a satisfactory balance has been achieved between increased energy requirements and increased breakdowns should other phases of a future study be undertaken.
2. Various packings should be tested for longer term operability using clean emulsions.
3. Packings should be tested in different diameter tubes operating under the same parameters to determine the magnitude of the wall effect with packings that have satisfactory long-term operability.
4. Continuous gravity separators of various configurations and materials should be tested using the various fuel emulsion candidates. This testing should be done with emulsion streams that have undergone a high degree of breakdown.

APPENDIX

PSEUDO-SHEAR RATE FORMULA

The pseudo-shear rate which has dimension of reciprocal time for flow through a circular passage covered with a screen is given by the equation below:

$$PSR = \frac{128}{\pi} \frac{Q_t}{(d\ N_{sr})^2(1/N_{sr} - D_{sr})^3}$$

where PSR is pseudo-shear rate

Q_t is total flow through the passage

d is the diameter of the passage

D_{sr} is the diameter of wire used in the screen

N_{sr} is the number of wires per unit length in the screen

When it is desired to obtain the pseudo-shear rate in reciprocal seconds, with the dimensions expressed in inches and the flow rate in gallons per minute, the equation becomes:

$$PSR = 50.0 \frac{Q_t}{(d\ N_{sr})^2(1/N_{sr} - D_{sr})^3}$$

For the same dimensions except with the flow expressed in milliliters per minute, the equation becomes:

$$PSR = 0.0132 \frac{Q_t}{(d\ N_{sr})^2(1/N_{sr} - D_{sr})^3}$$

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13. ABSTRACT

It is desirable to have a demulsification process so that fuel recovered from safety fuel emulsions could then be used in devices or vehicles not qualified for emulsion fuel. The technical feasibility of all three essential steps was demonstrated:

- Breakdown of emulsion
- Coalescence of unbroken emulsion
- Separation of unbroken emulsion from the fuel.

In this study, stainless-steel wool packing or screens were used for the critical steps of breaking and coalescing. Depending on the emulsions, 79 to 92% breakdown by weight was achieved. This performance was obtained by passing 1.4 gpm of the emulsions through a small tube (1/4 inch in diameter by 1-1/2 inches long) containing the steel wool. The most important parameter determining the extent of breakdown was found to be the energy dissipated per unit volume of emulsion. The best breakdowns were obtained at the upper limit of energy dissipation of the equipment used. If this energy is increased either by operating at pressures greater than 240 psi or by using a recirculating system, the extent of emulsion breakdown should be increased. Continuous separation of broken emulsion was demonstrated in this study using Esso WSX 7165 fuel emulsion. The resulting JP-4 met the military specifications (MIL-T-5624G) with the exception of dissolved surfactant, which conventional testing procedures classified as gum. Although the surfactant was removed by adsorption on charcoal, it is questionable whether it represents a significant debit. Since the scale-up of packed beds is well understood, further development could lead to either practical field depot units or small, vehicle-mounted demulsification units.

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