THICKENER/FLUID INTERACTION IN LUBRICATING GREASES

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By

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To allow a better understanding of the basic mechanisms of oil retention by grease thickeners, techniques were developed to extract the thickener from greases for microstructural study. An approach was developed to allow thickener coating of glass microbeads for use as column-packing media for High Performance Liquid Chromatography (HPLC) evaluation of the thickener's molecular adsorption effects with base oils.

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ABSTRACT, Continued.

Extensive testing of thickeners by HPLC under a variety of test conditions showed no resolvable changes in column retention times for different hydrocarbon types or base oil fractions. Considerable microstructural variation was detected between thickener types and thickeners of the same type from different manufacturers. This variation indicates that the effects of thickener microstructure on the containment of base oils by mechanical entrapment and capillary effects may have a greater influence on oil retention than the molecular adsorption mechanism.

FOREWORD

The work reported herein was conducted at the U.S. Army Fuels and Lubricants Research Laboratory (AFLRL), Southwest Research Institute, San Antonio, TX, under Contract Nos. DAAK70-80-C-0001 and DAAK70-82-C-0001 and covers the period March 1980 through October 1983. The work was funded by the U.S. Army Mobility Equipment Research and Development Command (MERADCOM), currently Belvoir Research and Development Center, Fort Belvoir, VA. Contracting officer's representative was Mr. F.W. Schaekel, Fuels and Lubricant Division/DRDME-GL (currently STRBE-VF), and the technical monitors were Drs. Herman Spitzer and Charles Chapin, Fuels and Lubricants Division/DRDME-GL (currently STRBE-VF), MERADCOM.

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I. INTRODUCTION

Lubricating greases have presented many problems in service and storage. Consistency often varies with time, causing the grease to either release oil from the thickener, or the entire mass may increase in thickness and even harden. Temperature extremes can affect greases by causing breakdown and bleedout at high temperatures, or thickening at low temperatures. Water interactions create problems by causing washout of grease from wheel bearings and chassis points, and water can also increase the problem of corrosion protection if the grease has poor preservative qualities. The inherent wear-reducing qualities and service life of greases are also of major concern.

These problem areas were identified in a world-wide survey of field units where the need for improved greases was established.(1)*

While problem areas encountered with greases have been known for many years, the basic mechanisms of the interactions of base oils with thickeners, and the effects these interactions have on serviceability of the greases are poorly understood.

Some works have suggested that base oil is contained by capillary effects and physiochemically by van der Wall's forces adsorption, etc.(2) Others have reported that oil separation phenomena can be described as a desorption process.(3)

Moniwa and Komatsuzaki felt oil which bleeds out was mechanically supported, and retained oil is physiochemically supported.(4) Considerable disagreement is present in reported literature as to actual microstructures of the thickeners. For example, many works describe Lithium 12-hydroxystearate thickeners as twisted strands.(5-7) Peterson and Bondi felt structural

* Underscored numbers in parentheses refer to the list of references at the end of this report.

damage was introduced by receding meniscus of the deoiling solvent during sample preparation for electron microscopy.(8)

Kistler used solvent extraction of the oil and critical point drying of the solvent to produce an aerogel of thickener, thus avoiding meniscus effects.(9) Anderson, et al. (10) deoiled small quantities of grease using a wicking action of solvent through filter paper and reported intact through dimensional structures without broken strands or twisting of the strands similar to results of aerogel technique. They concluded that twisted strands only occur if strands are broken from the structure by working of the grease or if structural damage is introduced by sample preparation techniques. Other investigators report the twist was present initially in the grease and not an artifact of sample preparation.(5-7) Vamos, et al. (11) reported broken strands with twisting following working of grease.

Studies of greases are further complicated by individual grease manufacturers having proprietary processes for fabrication, with variations of thickener types, base oil compositions, heating and cooling rates, and mechanical process parameters. This can cause considerable variation in greases from different manufacturers and some variation from batch-to-batch with any one manufacturer.

The purpose of this program was to investigate the interactions of grease thickeners with base oils and to more clearly define the mechanisms of these interactions.

II. APPROACH

The retention of base oil by the thickeners is thought to be due to molecular adsorption of the oil by the thickener, mechanical entrapment of the oil by the thickener, and capillary effects of the thickener.

The main thrust of this study was to evaluate molecular adsorption as a mechanism, and High-Performance Liquid Chromatography (HPLC) was selected as a possible approach to evaluate molecular adsorption.

Scanning Electron Microscopy (SEM) was to be utilized in evaluating the quality of thickener-coated glass microbeads for the packing of columns to test by HPLC. The SEM was also to be used to document the thickener microstructures encountered during the program to determine if variations in microstructure could account for differences in grease behavior.

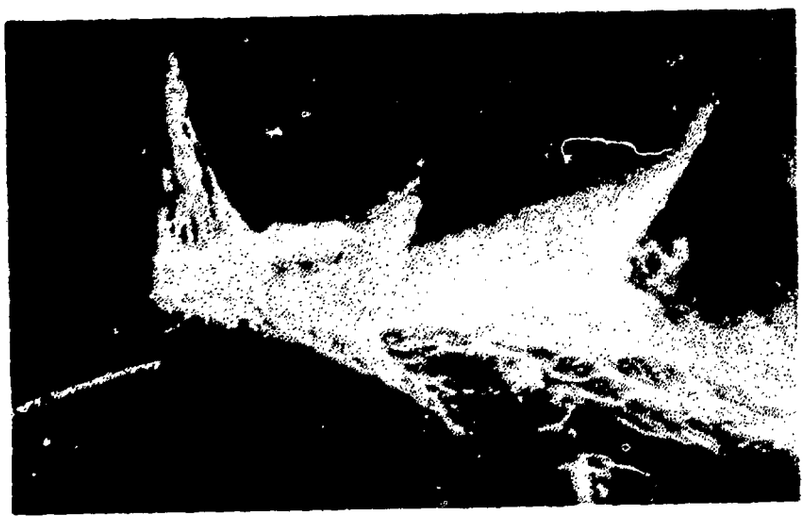
III. HPLC TESTING FOR MOLECULAR ADSORPTION OF OILS BY THICKENERS

In order to test the grease thickeners by High-Performance Liquid Chromatography, it was necessary to develop techniques to extract the base oil from the grease and apply the remaining thickener material to support media for packing into the HPLC column.

Glass microbeads 37 micrometers in diameter were selected as the supporting media, and grease containing lithium 12-hydroxystearate as thickener was chosen for extraction. Loading ratios were chosen to produce coating thicknesses of 0.4 percent, 4 percent, and 40 percent of thickness to bead diameter. The base oil was extracted from the mixture using n-heptane in a soxhlet extractor. The extracted oil was recovered for future use, and the glass microbeads with thickeners were tumbled and dried in a rotary evaporator. Although this technique produced encouraging results, it obviously needed refinement since the coatings were somewhat uneven and had a tendency to peel away from the microbead as shown in Figure 1. At the high magnification of Figure 1(c), individual strands of the coating could be distinguished with the characteristic twisted appearance that had been noted in earlier work.(5-7,12-14) The areas of the microbeads that were heavily coated appeared to be a tightly compacted mat of fibers.

In an attempt to produce a more evenly distributed coating on the microbeads, the grease was prediluted with n-heptane before the microbeads were mixed into the grease. The mixture was then placed in the soxhlet extractor and the oil removed using n-heptane. After using this approach with a variety of loading ratios, scanning electron microscope examination of the coated beads determined that only very low-loading ratios (<4 percent) could be evenly applied using this technique. All attempts at higher loading ratios produced uneven coatings with a tendency to peel away from the beads, similar to the condition shown in Figure 1.

a. 500X



b. 5000X

20,000X

FIGURE 1. SCANNING ELECTRON MICROSCOPE PHOTOGRAPHS OF GLASS MICROBEADS WITH THICKENER

Freeze drying was selected as another approach to thickener coating of the microbeads. The grease was shaken with benzene to dissolve the oil and was then gently centrifuged to collect the thickener at the bottom of the centrifuge tube. The thickener was then shaken with benzene and centrifuged two more times to ensure that the base oil had been totally removed. Following the final centrifuging, the excess solvent was poured off, and the gel-like mixture of thickener and solvent was shaken with glass microbeads to suspend the microbeads in the gel. The mixture was then frozen and a vacuum applied to remove the solvent by sublimation. This process allowed the removal of the solvent without damage to the thickener structure that normally is caused by the concentrating effect and the surface disturbances in an evaporating liquid phase. The technique produced an extremely well-preserved thickener structure with coating of the beads and a very intricate three-dimensional filament-like network bridging between the microbeads. This structure is illustrated in Figure 2(a). It was noted in examining this structure that the "twisted rope" shape of the strands, as shown in Figure 1(c), and as reported in earlier works (5-7,12-14) with lithium 12-hydroxystearate thickeners, was not present in the well-preserved, three-dimensional matrix of thickener that was produced by the freeze drying technique. This tends to indicate that the twisting, which is often seen in strands of thickener prepared by other means, may be artifacts of the breaking of strands from the matrix or artifacts created by drying from a liquid phase, as has been suggested by other investigators.(8-10)

A small batch of thickener was then extracted from the grease and freeze dried without microbeads. SEM examination of lumps of the freeze dried thickener confirmed the same structure as was noted between the beads in Figure 2(a). Base oil which had been recovered from earlier extractions was then slowly added to the batch of freeze dried thickener to determine if it would reconstitute into a grease. As the drops of oil were added, they were rapidly absorbed by the thickener matrix. The absorption appeared visually to resemble a "wicking" action. Following addition of the base oil, the thickener formed a grease-like mass that was visually indistinguishable from the original grease in texture or color. This apparent ability to reconstitute indicates that the thickening ability of the material had not been affected by the freeze drying extraction method.

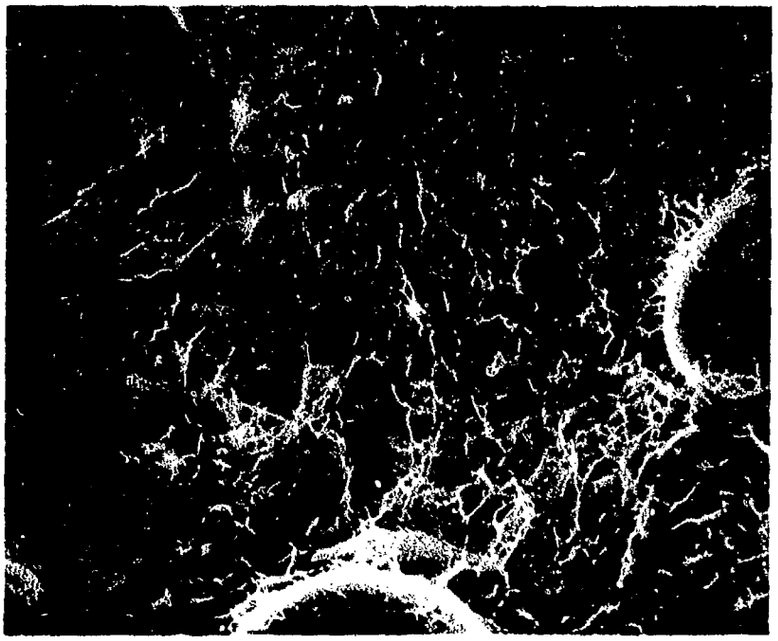


FIGURE 2(a). FILAMENT NETWORK BETWEEN BEADS (1000X)

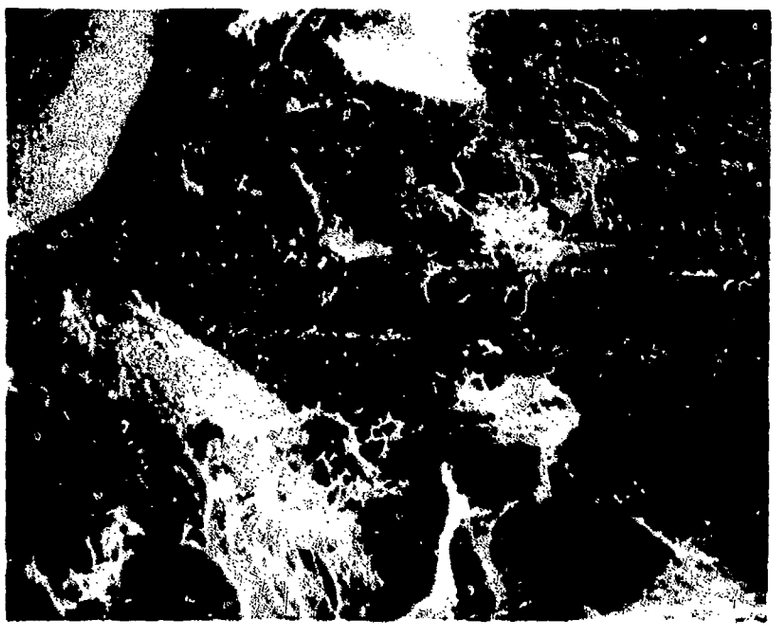


FIGURE 2(b). BROKEN STRANDS IN BEAD NETWORK FROM HPLC SOLVENT FLOW (2000X)

Another observation from this experiment is that the gel of solvent and thickener produced from the extraction process occupies four to five times the volume of the original grease. When the base oil is added to the freeze dried thickener, it returns to its original volume. If the reconstituted grease is once again extracted, the resulting structure is the same as was noted on the original grease extraction. This ability of the thickener matrix to change in volume while maintaining its structural network intact seems to be due to expansion or contraction of the entire matrix, depending on the type of fluid present. Whether or not the strands increase in diameter as they shorten in length to cause the volume changes of the matrix noted with oil versus benzene could not be determined within the scope of the present work.

Experiments were then conducted using n-heptane for the initial oil extraction and cyclohexane for the final extractions and freeze drying. Cyclohexane was chosen because of its relatively high freezing point and its lower toxicity. The results were the same as benzene freeze drying, so cyclohexane was used as the freeze drying solvent in all following tests.

To allow HPLC testing of the thickeners, it was necessary to place the thickener and supporting bead into HPLC columns. This was accomplished initially by forcing a slurry of the thickener and microbeads with cyclohexane into the column under pressure until the slurry filled the column. Samples of the slurry, as packed into the column and as removed from the column after testing, would be freeze dried for SEM evaluation of the structures. Testing of the slurry-packed columns produced peaks with good symmetry, indicating that the packing had been accomplished without introducing any voids into the column. However, back pressure began to build to a level too high to allow checks of retention times. When the column was disassembled and the packing freeze dried and examined, it was noted that the strands of the network between the beads had been broken by the flow of the carrier solvent through the column, as shown in Figure 2(b). Fragments from the broken thickener strands had plugged the fritted disc at the end of the column, causing the abnormal increase in back pressure.

It was determined that the interconnecting network of thickener strands between the microbeads could be eliminated by reducing the thickener to microbead-loading ratio and freeze drying the microbead/thickener/solvent mixture prior to column packing. The loading ratio which seemed to work best for the solvent extraction operation was 1 gram of bulk grease to 1 gram of microbeads. Following the freeze drying step, SEM evaluation of the microbeads revealed well-coated microbeads without interconnecting strands, which were suitable for dry packing into HPLC columns, as shown in Figure 3.

A quantity of 37- to 44-micron glass microbeads were coated with lithium 12-hydroxystearate thickener using the freeze drying technique and dry packed into HPLC columns 3 feet in length. A second column containing uncoated microbeads was also prepared to allow comparison with the coated bead column. Acetonitrile was selected as the carrier solvent, and both columns were purged with this solvent prior to testing.

The extracted base oil and several reagent-grade saturated and aromatic hydrocarbons were injected into the control column of uncoated microbeads, and time from injection to detection of each compound was recorded. The column retention time for the uncoated microbead column was about the same for each compound injected. This was as expected since no thickener was present to selectively retard the flow of the compounds through the column.

The same hydrocarbon compounds were then injected into the thickener-coated microbead column and retention times recorded. The initial results were encouraging in that a slight increase in retention time was noted as compared to the control column. The shift in retention time proved, however, to be the same for all the tested compounds and the base oil. This indicates that the shift in retention time was not due to variation in affinity between the various chain-length hydrocarbon compounds and the thickener. The slight increase in retention time was most likely due to the increased effective surface area of the coated microbeads as compared to the smooth surfaces of the uncoated microbeads.

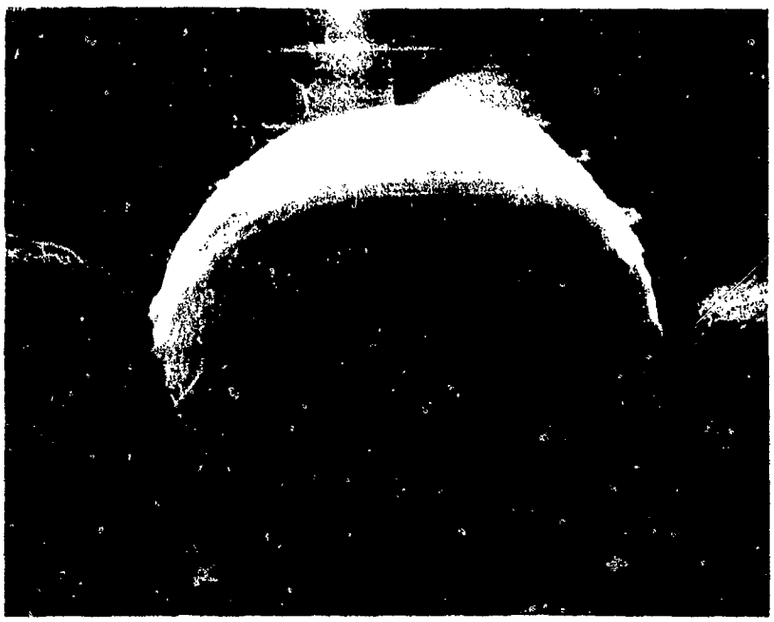


FIGURE 3. MICROBEADS PREPARED FOR HPLC COLUMN PACKING USING FREEZE DRYING TECHNIQUE FOR THICKENER COATING (2000X)

Since HPLC testing can be quite sensitive to the type of carrier solvent used, columns were prepared to allow testing of a variety of carrier solvents. Testing was performed using each of the following carrier solvents in both control and test columns:

1. Acetonitrile
2. Heptane
3. Hexane
4. Cyclohexane
5. Benzene
6. Toluene
7. Methanol
8. Tetrahydrofuran
9. Isopropyl alcohol
10. Fluorinated solvents
11. Chloroform

In all of the tests with the various solvents, no variation in retention times could be established when the test compounds were injected. To establish if variation in retention time was present but too slight to detect with the 3-foot column length, several 12-foot columns were constructed and tested, but variation of retention times still could not be detected. Attempted variations in carrier solvent flow rates and injected sample volume also failed to improve resolution.

It was then proposed that smaller diameter glass beads and/or reverse phase HPLC using solvent water combinations could be alternate approaches to improved resolution. Smaller microbeads proved to be an unacceptable approach due to high cost and limited availability. The reverse phase approach was tested using isopropyl alcohol with water additions of from 2 to 10 percent to determine effects on column retention times. The addition of water to the carrier solvent proved unsuitable due to the thickener having some water solubility. Fragments of the coating were washed from the beads, causing blockage of the fritted disc at the output end of the column. This effect was noted even with water concentration as low as 2 percent.

Since oil retention properties of a thickener are attributable to both adsorption on the molecular level and mechanical entrapment on the microstructural level, an extensive evaluation of microstructural characteristics was to be conducted within this program. The difficulties encountered in detecting molecular adsorption using HPLC caused a shift of major effort to microstructural evaluation for the remainder of the program.

IV. MICROSTRUCTURAL EVALUATION OF GREASE THICKENERS

To allow documentation and characterization of a variety of grease types, 69 grease samples were selected by Belvoir R&D Center for microstructural examination. A listing of these greases is shown in Table 1. All the greases were evaluated with the exception of sample 37, which proved difficult to extract, and sample 46, which was not received in the shipment of greases.

For preparation of thickener specimens, procedural development was required. The selected procedure was to remove small specimens of grease from 10 randomly selected areas of the grease sample. The 10 specimens were then combined, and the oil removed by solvent extraction. The final solvent extraction was performed using cyclohexane, and the gel was freeze dried as in the HPLC extraction process. The freeze dried specimen was gently mixed and a portion removed for mounting on a SEM stub. The mounted specimen was then given a thin vacuum evaporated coating to improve electrical conduction for SEM examination. Each prepared sample was scanned in the SEM, and an area typical of the structure encountered was selected for photographic documentation.

While a wide variation in microstructure types was encountered in this program, there were sufficient similarities between calcium, lithium, barium, sodium, and mixed-base greases to allow them to be discussed as a group. Microstructures for this group of thickeners are illustrated in Figures 4 through 13.

One of the more commonly encountered structures within this group of thickeners is the interconnected three-dimensional network of strands shown in Figures 4 through 6. While the size of this matrix seems to vary with thickener type and manufacture, the basic networks are quite similar. Since the photographs presented are quite high in magnification, it is easy to envision that the close spacing of the strands could easily cause entrapment of the oil by mechanical and/or capillary effects in this type of structure. Figures 7 through 13 represent variations from network of strands shown in

TABLE 1. GREASE SAMPLES SELECTED FOR MICROSTRUCTURAL EXAMINATION

Sample No.	Base Oil	Thickener	Comments
1	PAO	Polyurea	
2	Mineral	Calcium	
3	Mineral	Mixed Base	New
4	Mineral	Mixed Base	Old
5	Mineral	Mixed Base	
6	Mineral	Barium	
7	Mineral	Barium	
8	Mineral	Barium (Moly)	
9	Mineral	Bentone Clay	New
10	Mineral	Bentone Clay	Old
11	Mineral	Bentone Clay	New
12	Mineral	Bentone Clay	Old
13	Mineral	Li	
14	Mineral	Polyurea Acetate	New
15	Mineral	Calcium	
16	Ester	Sodium, Organic Dye	
17	Mineral	Sodium	
18	Ester	Microgel	MIL-G-23827
19	Mineral	Calcium	
20	Mineral	Calcium	
21	Mineral	Ca/Li	
22	Mineral	Ca/Li	
23	Mineral	Li	
24	Ester	Clay	New
25	Ester	Clay	Old
26	Ester	Clay	
27	Ester	Clay	Old
28	Ester	Clay	
29	Mineral	Calcium	

(Continued)

TABLE 1. GREASE SAMPLES SELECTED FOR MICROSTRUCTURAL EXAMINATION (CONT'D)

Sample No.	Base Oil	Thickener	Comments
30	PAO	Polyurea	
31	---	---	
32	---	---	
33	Ester	Li (Moly)	MIL-G-21164
34	Silicone	Li	MIL-G-4343
35	Mineral	Li	
36	Silicone	---	
37	Silicone	---	
38	PAO	Clay	DOD-G-24508
39	Mineral	Sodium or Calcium	VV-G-632A
40	Mineral	Microgel	MIL-G-24139
41	PAO	Clay (Moly)	MIL-81827
42	Mineral	Calcium	MIL-G-25537
43	Silicone	Moly	MIL-G-4343
44	Silicone	Organic Dye	MIL-G-25013
45	Ester	Polyurea	MIL-G-23549
46	Ester	Lithium (Moly)	MIL-G-21164
47	Mineral	Li (Teflon)	
48	Mineral	Lithium	MIL-G-18209
49	Mineral	Lithium	MIL-G-18209
50	PAO	Clay	MIL-G-81322
51	Mineral	Li (Teflon)	
52	Mineral	Calcium	
53	PAO	Lithium Complex	
54	Mineral	Lithium or Ca	
55	---	---	MIL-G-83363
56	Ester	Clay	
57	Ester	Clay or Li	MIL-G-23827
58	---	---	

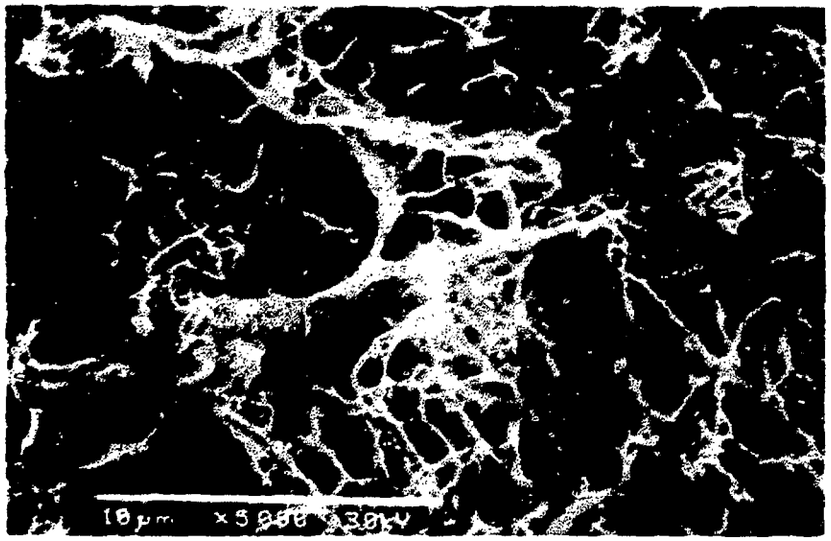
(Continued)

TABLE 1. GREASE SAMPLES SELECTED FOR MICROSTRUCTURAL EXAMINATION (CONT'D)

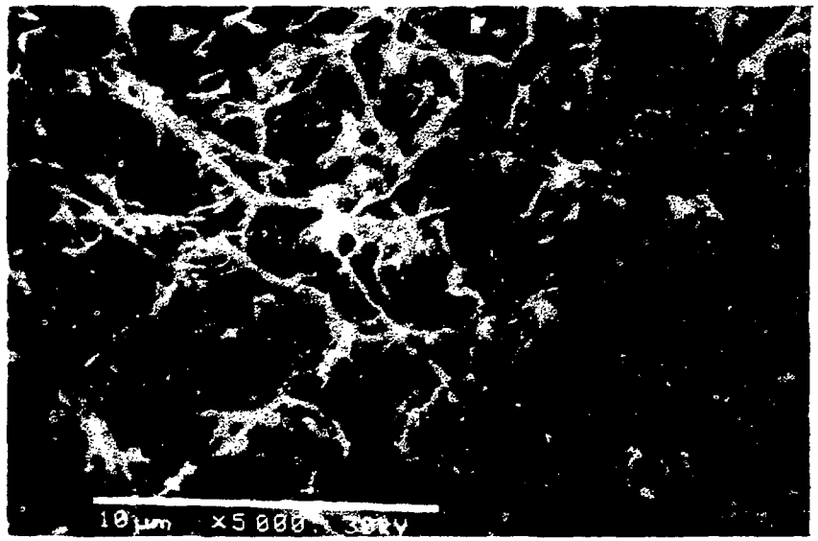
Sample No.	Base Oil	Thickener	Comments
59	PAO	---	
60	Mineral	Thickener	
61	Mineral	Polyurea	
62	Mineral	Lithium	
63	Mineral	Lithium	MIL-G-10924D
64	Mineral	Lithium	
65	Mineral	Li Complex	
66	PAO	Polyurea	700 miles
67	Mineral	Li or Ca	300 miles
68	Mineral	Li/Ca	Outer Bearing, 1000 miles
69	Mineral	Li/Ca	Inner Bearing, 1000 miles

earlier figures. These structures vary from a lumpy amorphous-looking mass, such as shown in Figures 7(c) and 9(d), to partially resolved networks or compacted strands, such as shown in Figure 8(b) and 8(c). Two of the structures shown from this group are totally different from any of the others. Sample 15 in Figure 13(c) has a short rod-like structure which more closely resembles the polyurea thickeners, and sample 64 in Figure 13(d) more closely resembles the clay type thickeners.

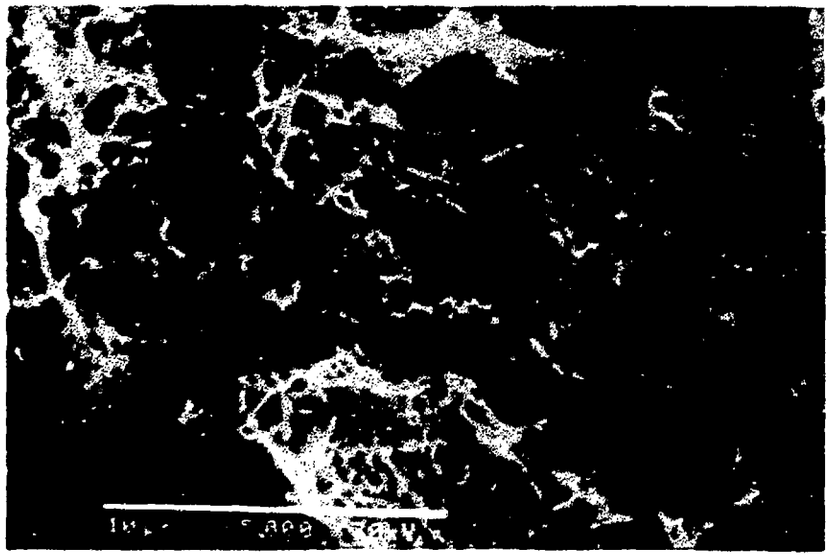
Another group of thickeners examined in this program were the clay thickeners whose microstructures are shown in Figures 14 through 17. The first two microstructures illustrated in this group, Figures 14(a) and 14(b), are somewhat suspect as to whether the identification of them as clays is correct since they possess the strand-like network noted in the earlier group of structures. The majority of the clay microstructures can be described as laminate-appearing platelets, such as in Figures 14(d) and 16(d), agglomerated lump as in Figure 17(a), or a mixture of these two primary microstructures.



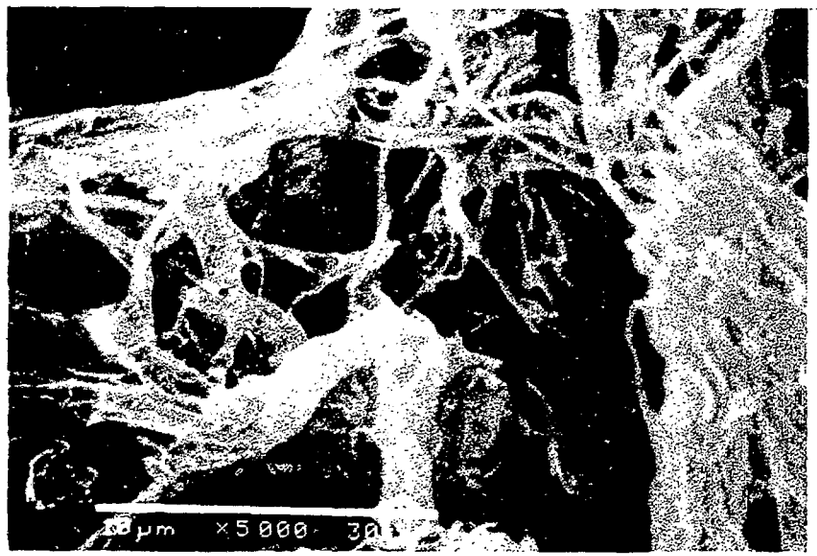
(a) Sample 13 Lithium



(b) Sample 16 Sodium

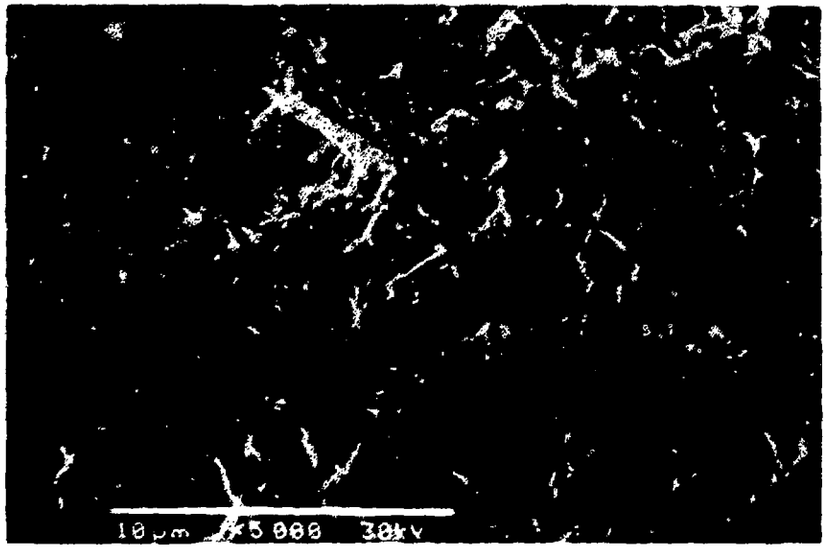


(c) Sample 21 Ca/Li

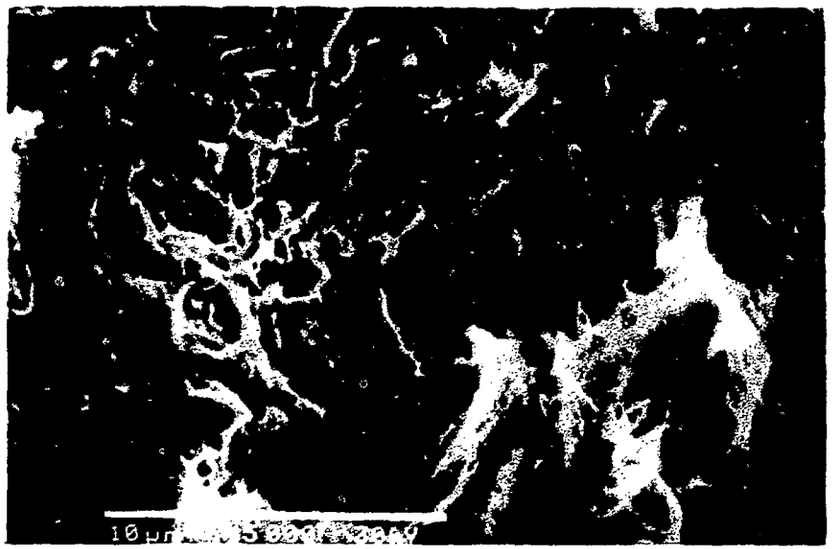


(d) Sample 8 Barium

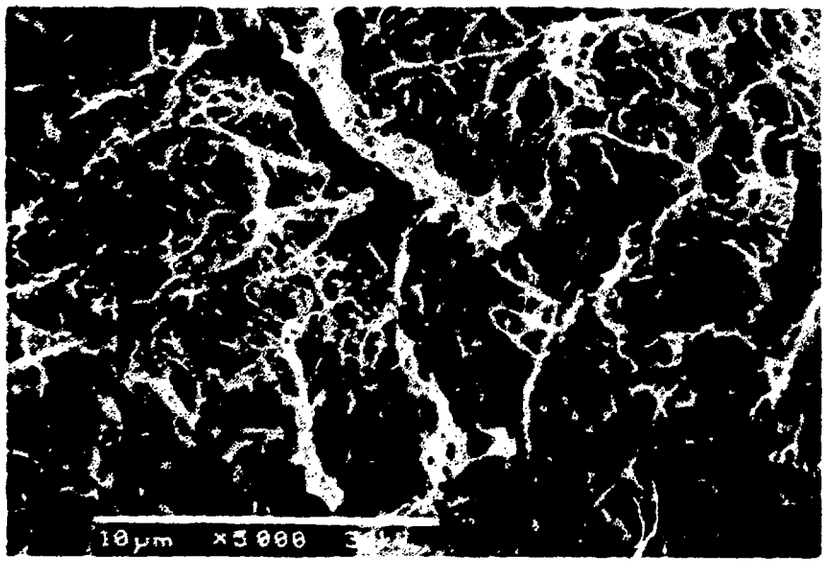
FIGURE 4. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 8, 13, 16 & 21



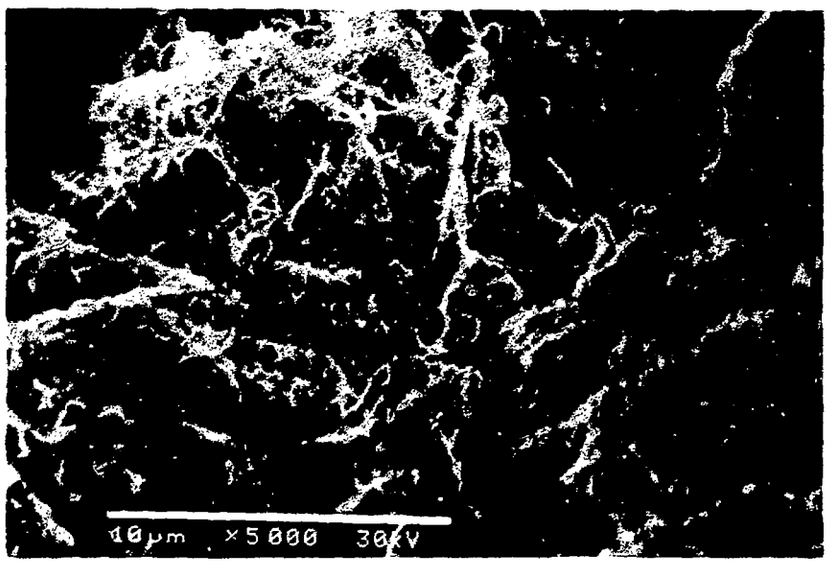
(a) Sample 60 Unidentified



(b) Sample 17 Sodium

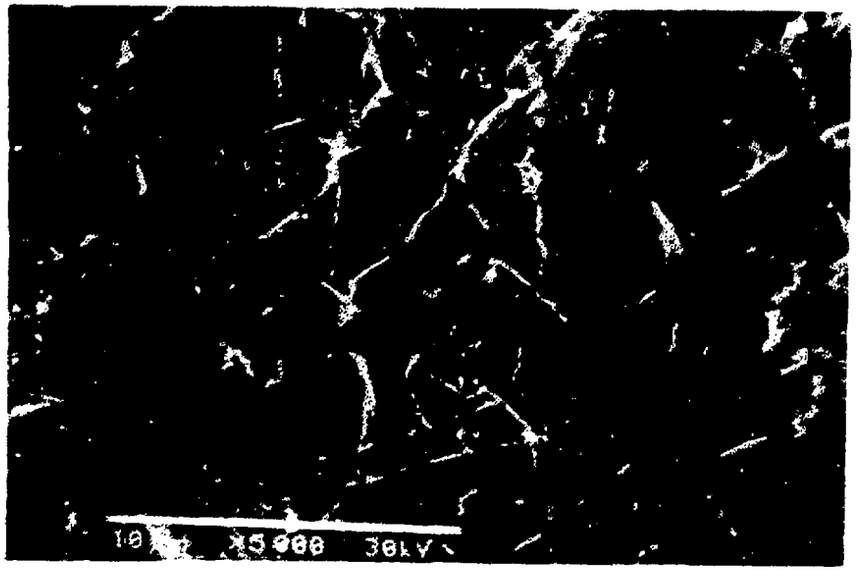


(c) Sample 65 Lithium

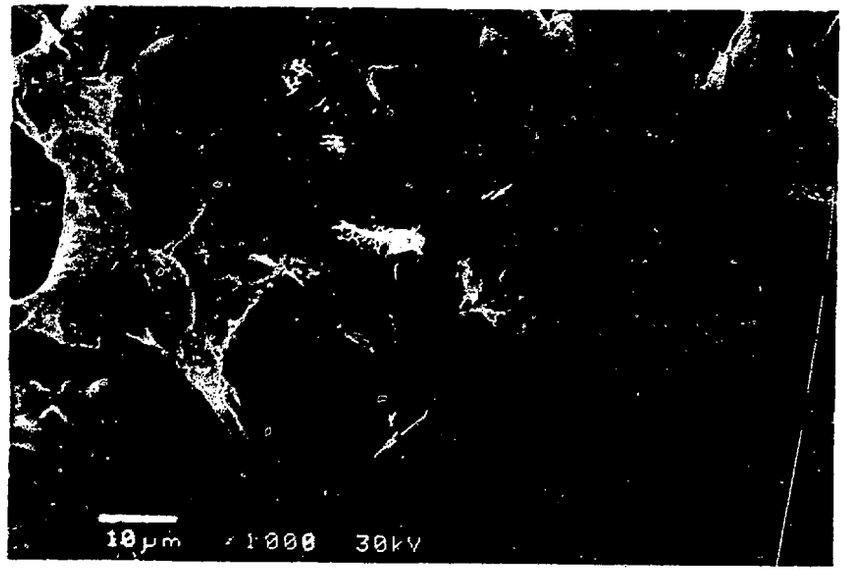


(d) Sample 62 Lithium

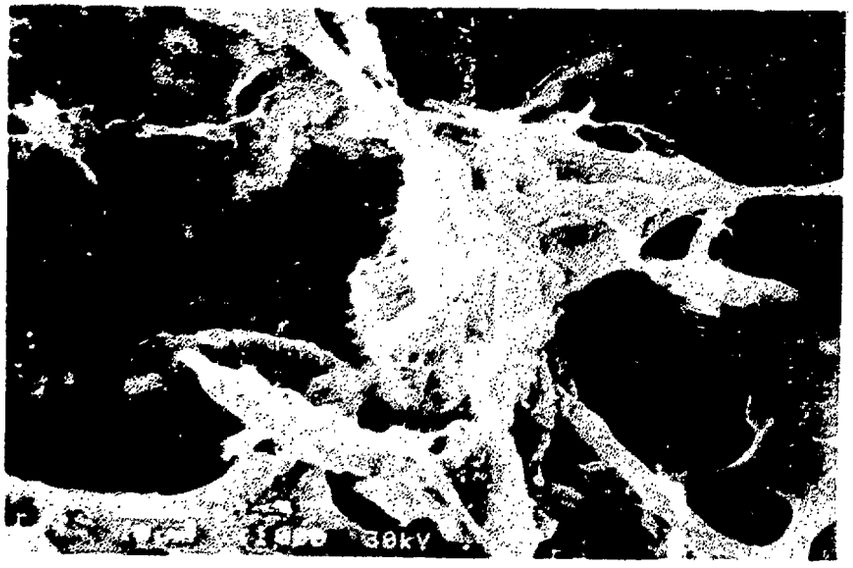
FIGURE 5. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 17, 60, 62 & 65



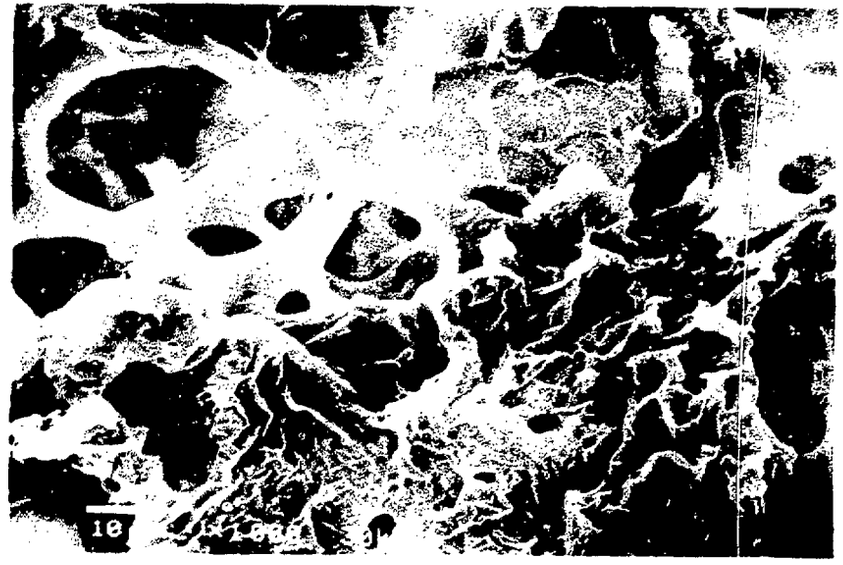
(a) Sample 53 Lithium



(b) Sample 19 Calcium

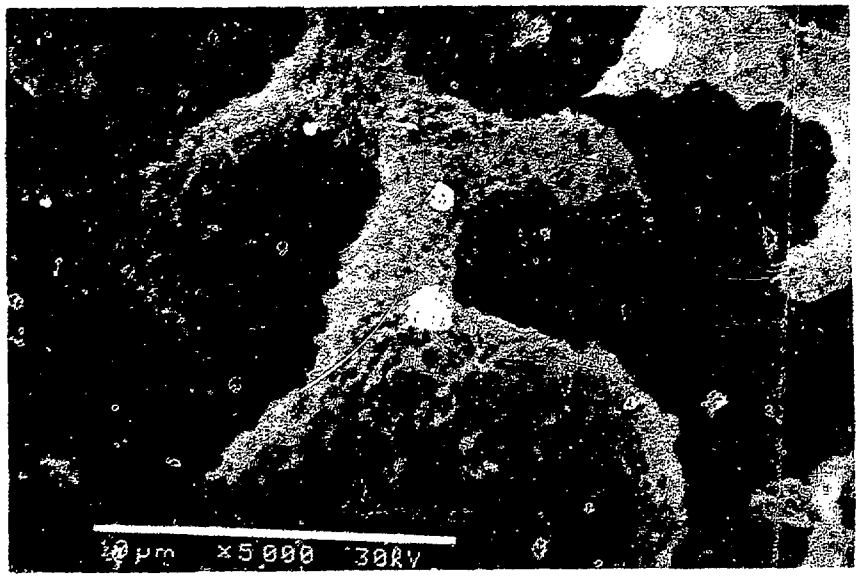


(c) Sample 6 Barium

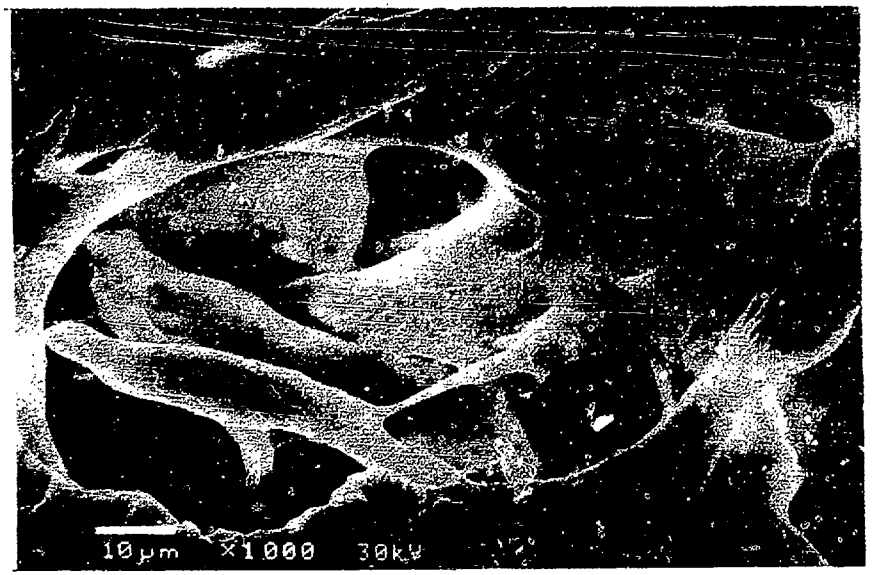


(d) Sample 6 Barium

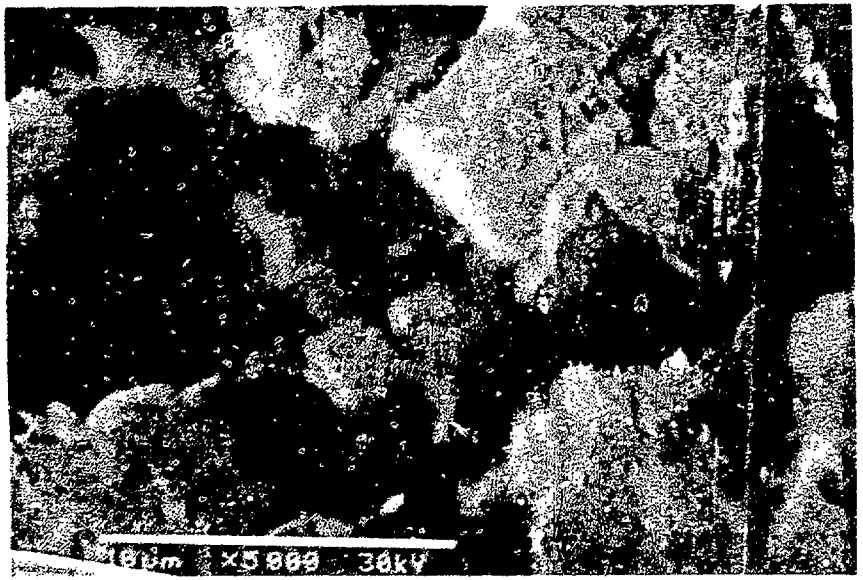
FIGURE 6. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 6, 19 & 53



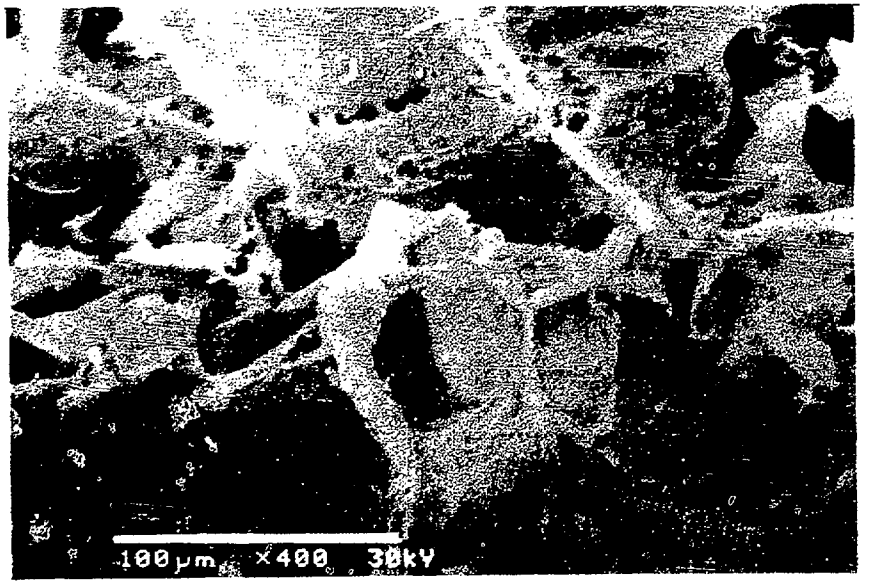
(a) Sample 51 Lithium



(b) Sample 42 Calcium

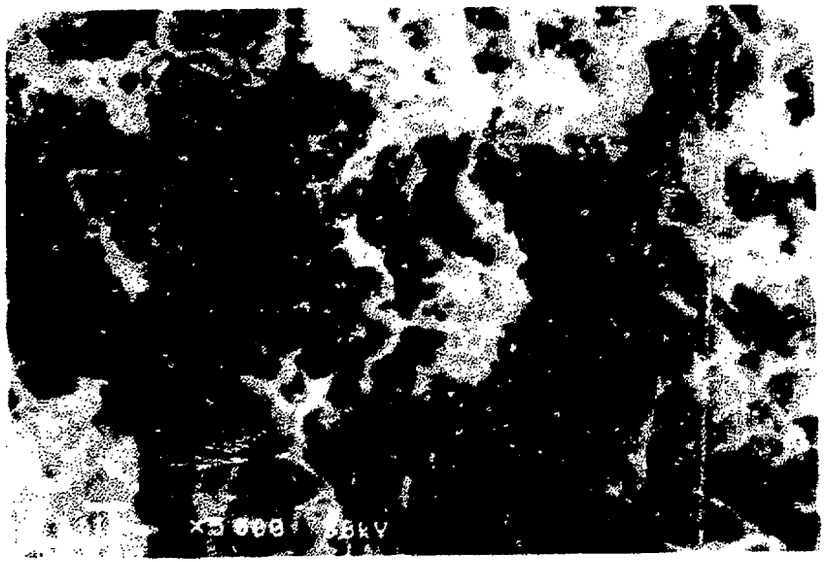


(c) Sample C61 Li or Ca

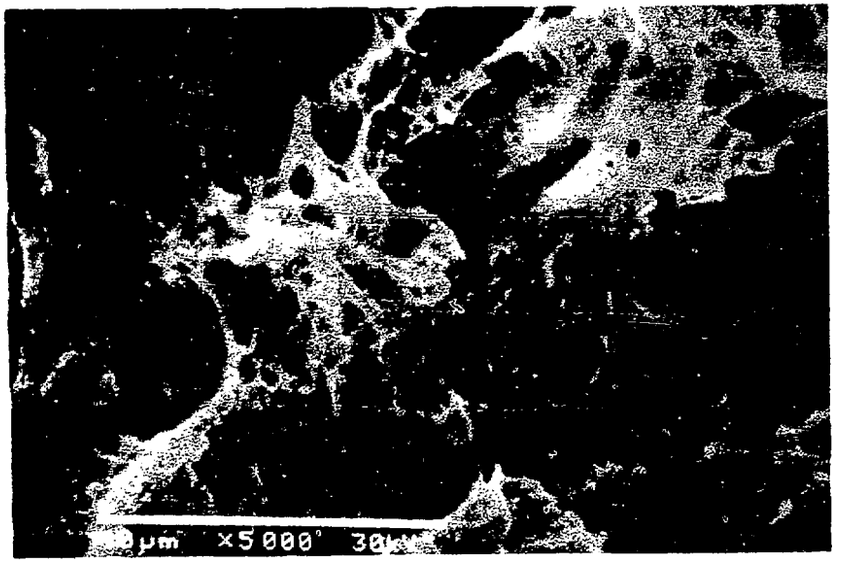


(d) Sample 4 Mixed base

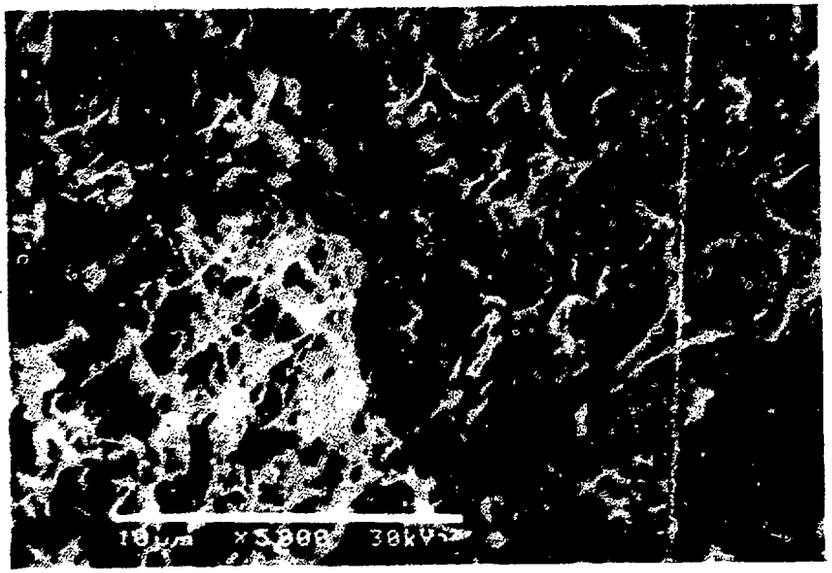
FIGURE 7. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 4, 42, 51 & C61



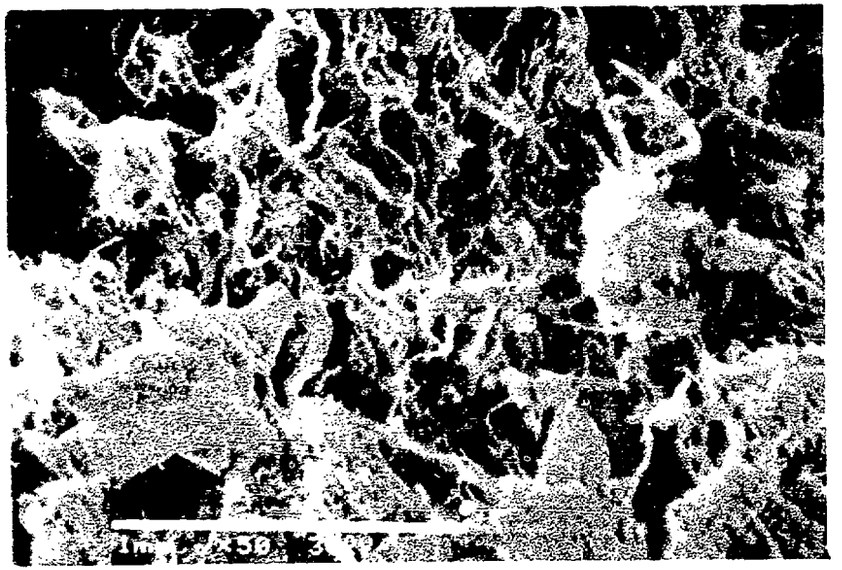
(a) Sample 3 Mixed base



(b) Sample 22 Ca/Li

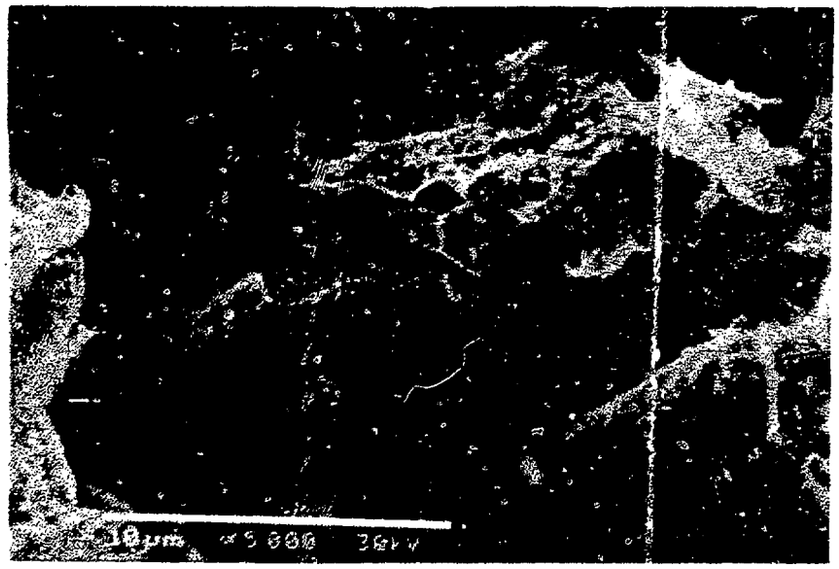


(c) Sample 52 Calcium

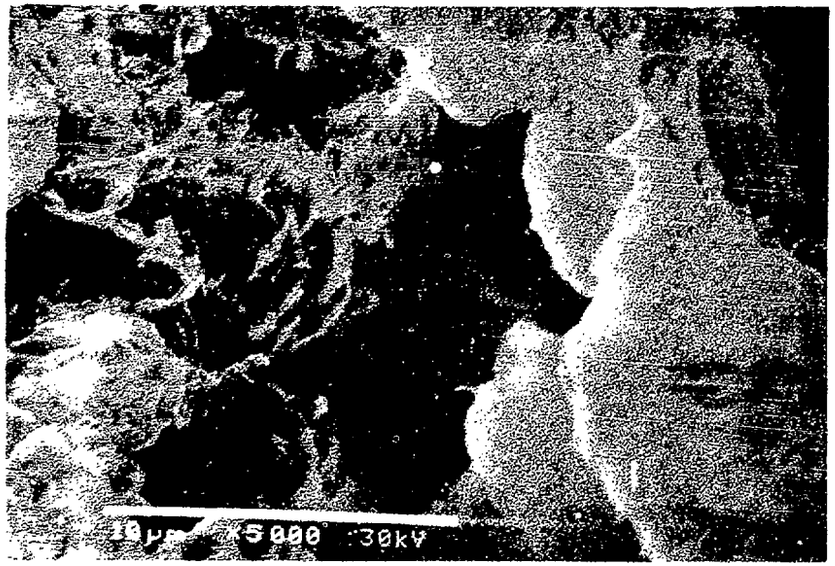


(d) Sample 23 Lithium

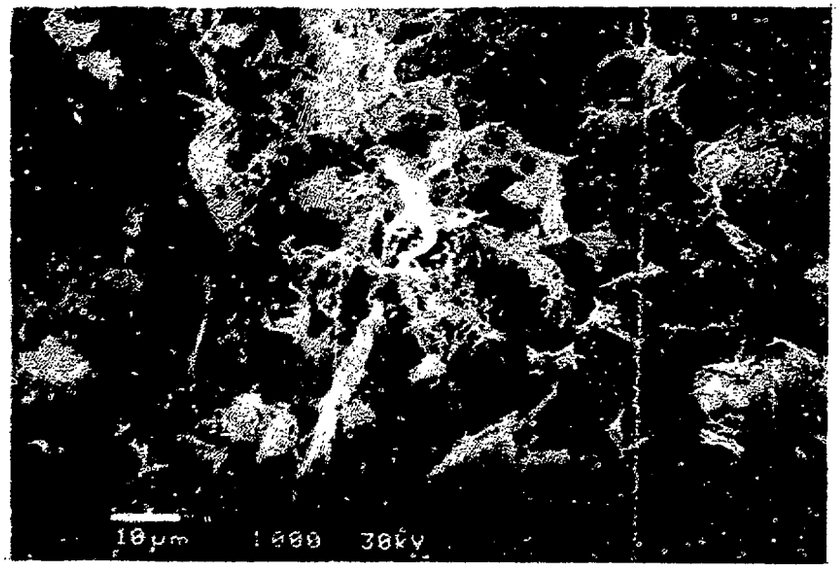
FIGURE 8. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 3, 22, 23 & 52



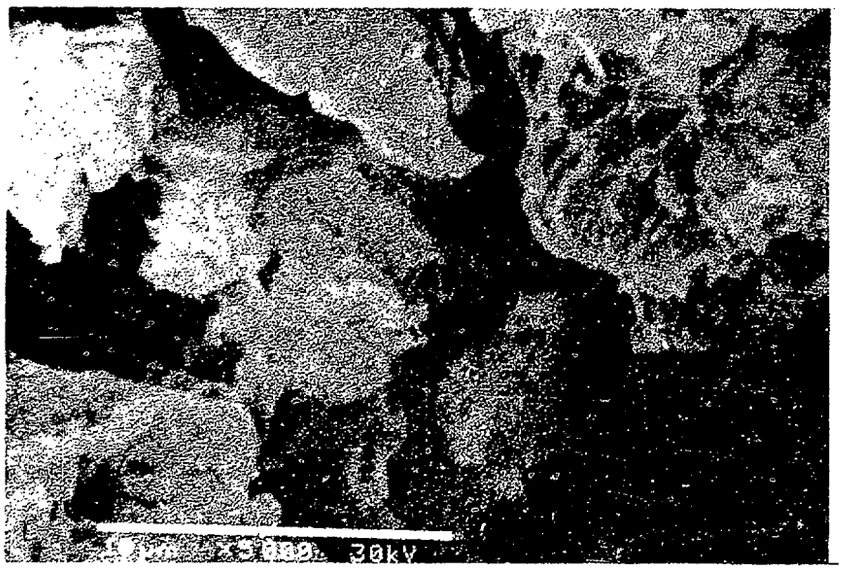
(a) Sample 20 Calcium



(b) Sample 48 Lithium

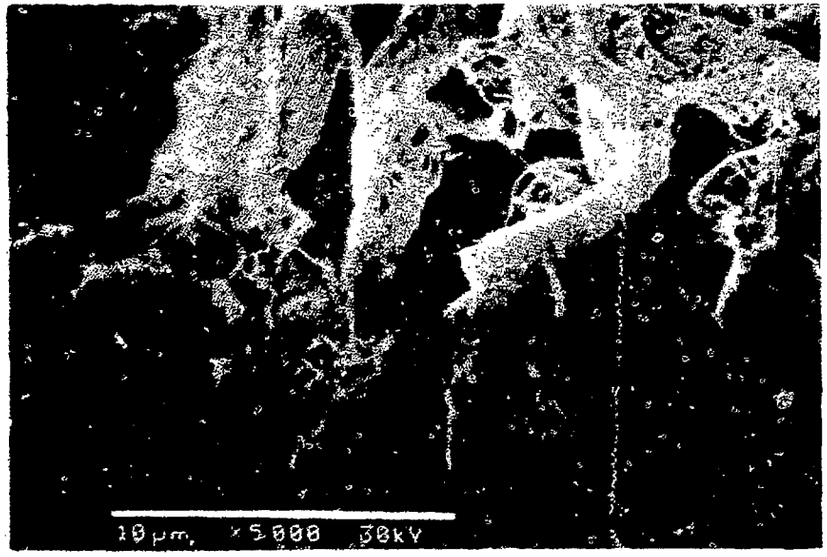


(c) Sample B61 Li/Ca
Inner bearing

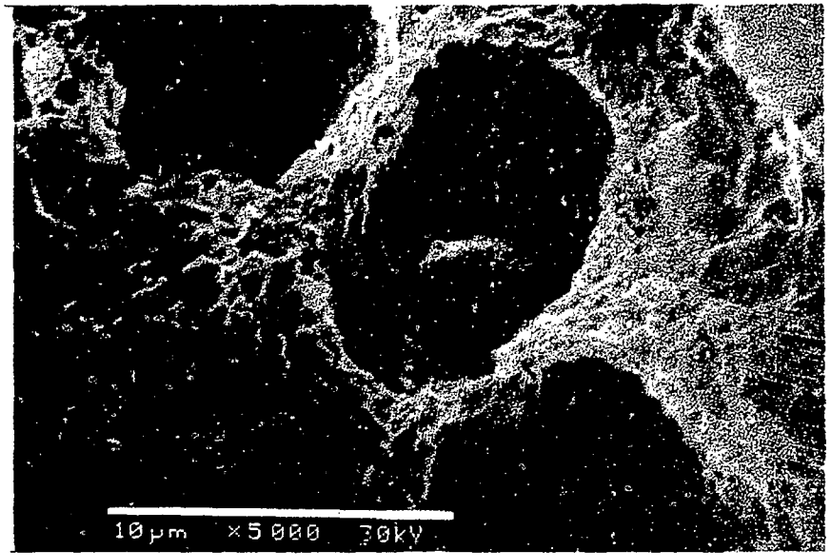


(d) Sample B61 Li/Ca
Outer bearing

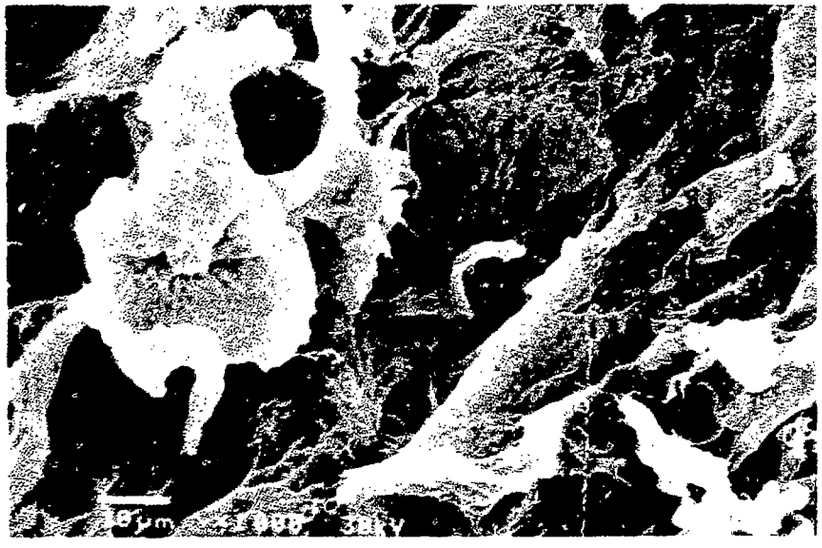
FIGURE 9. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 20, 48 & B61



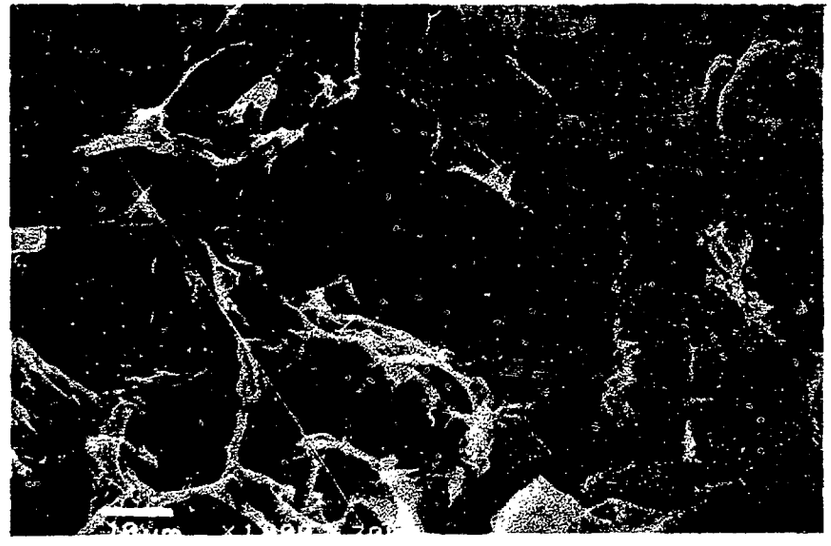
(a) Sample B61 Lithium
Inner Bearing



(b) Sample 49 Lithium

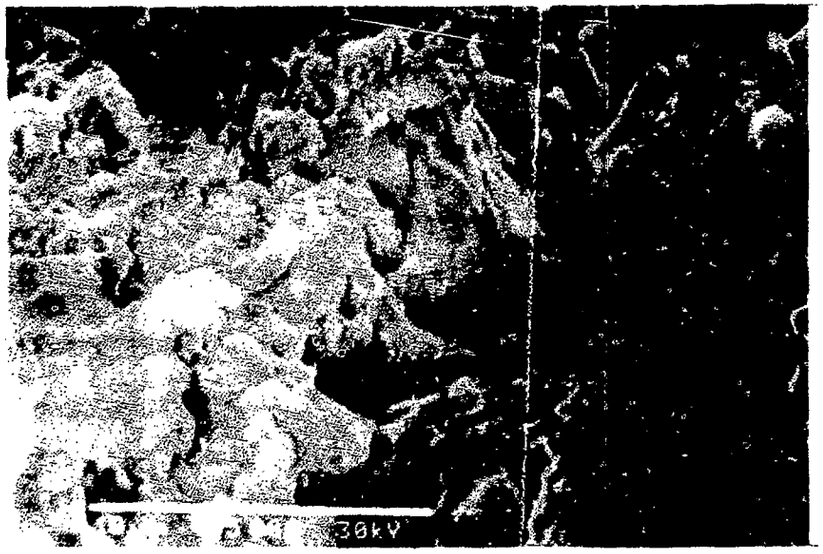


(c) Sample 2 Calcium

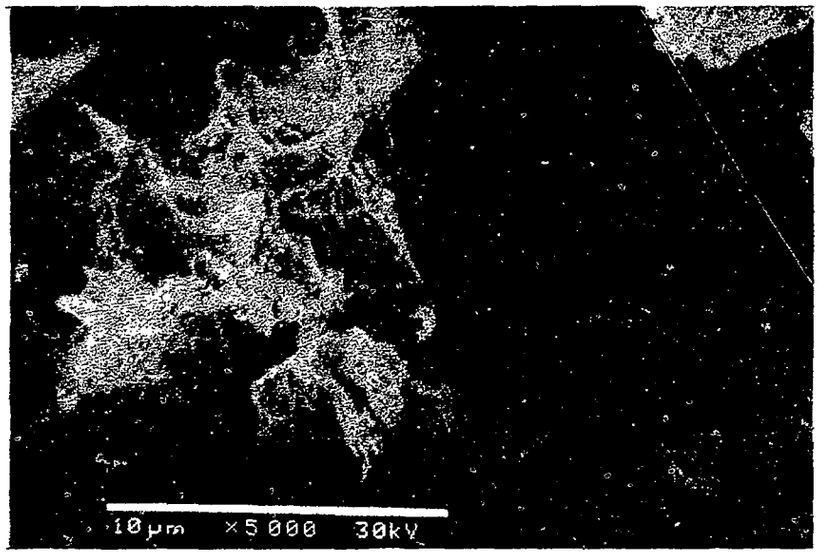


(d) Sample 19 Calcium

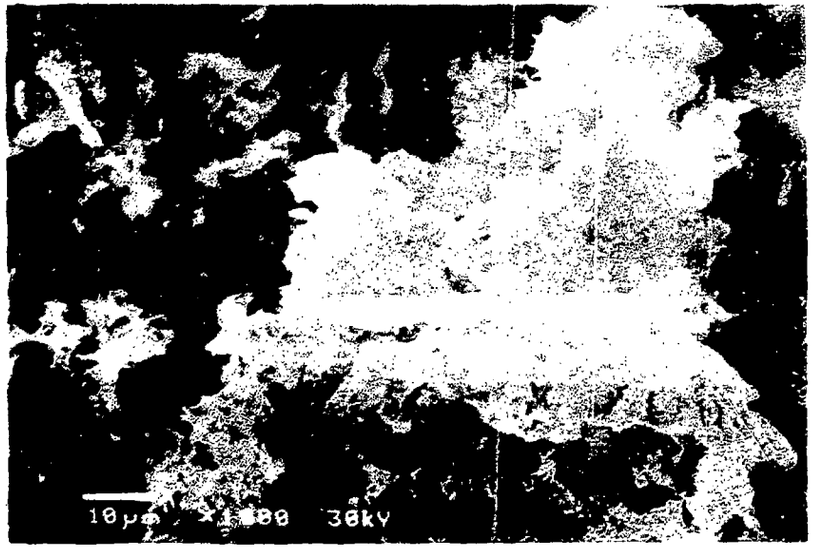
FIGURE 10. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 2, 19, 49 & B61



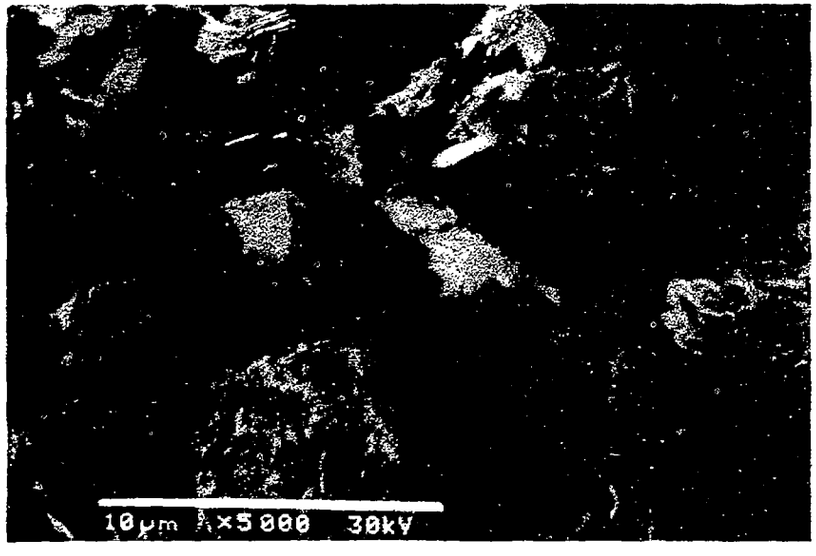
(a) Sample 35 Lithium



(b) Sample 63 Lithium

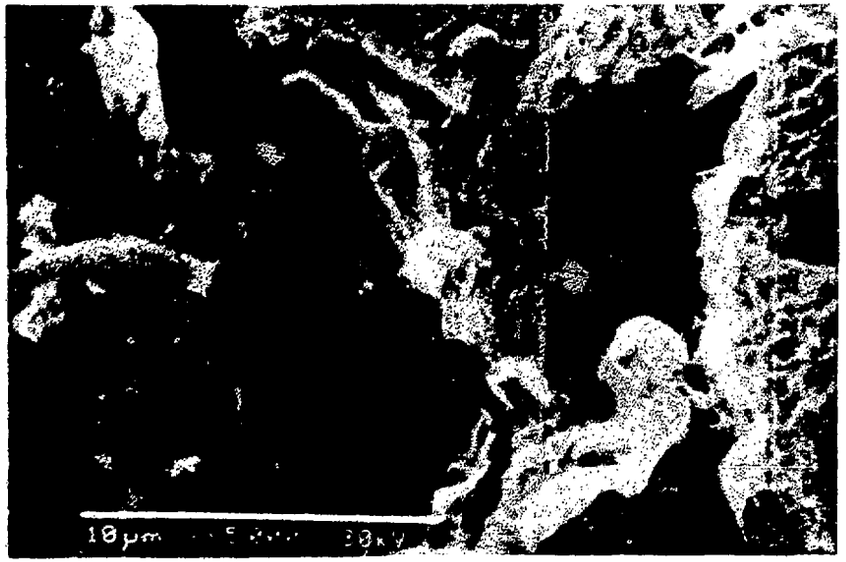


(c) Sample 5 Mixed Base

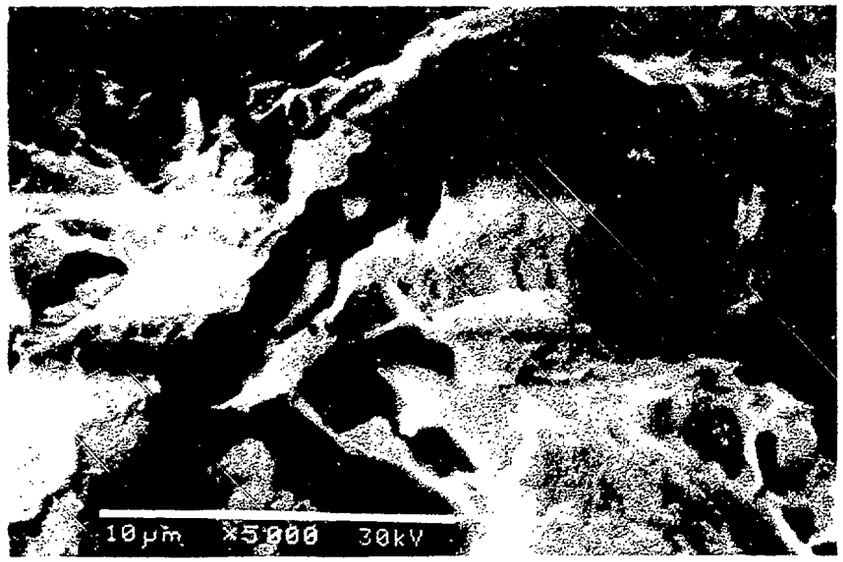


(d) Sample 33 Lithium

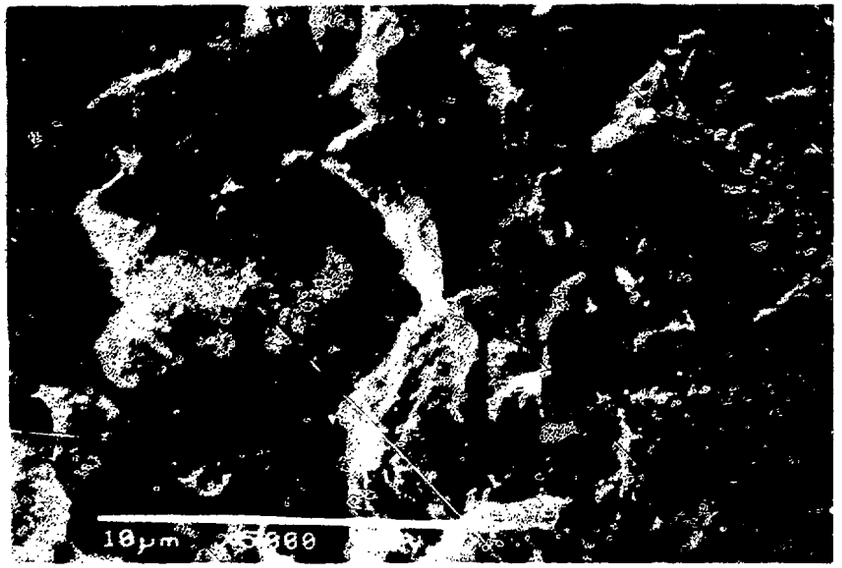
FIGURE 11. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 5, 33, 35 & 63



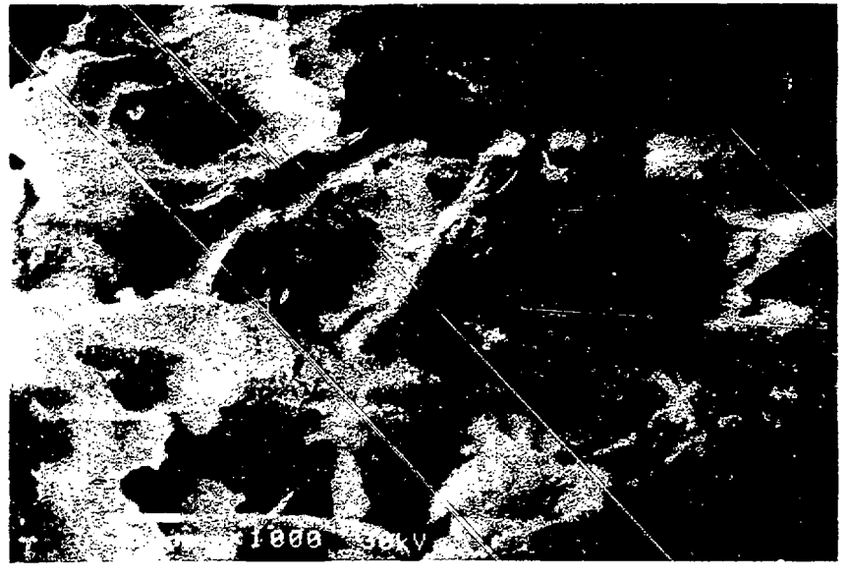
(a) Sample 39 Na or Ca



(b) Sample 34 Lithium

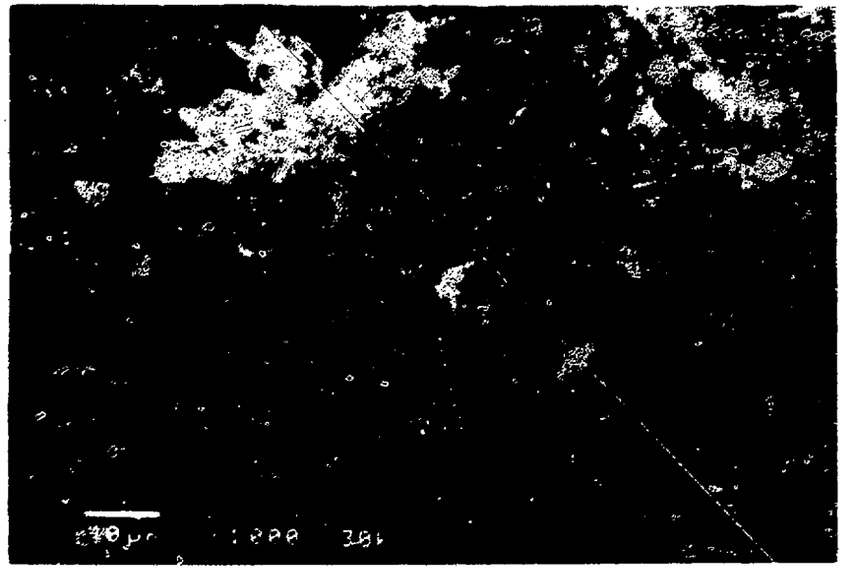


(c) Sample 54 Li or Ca

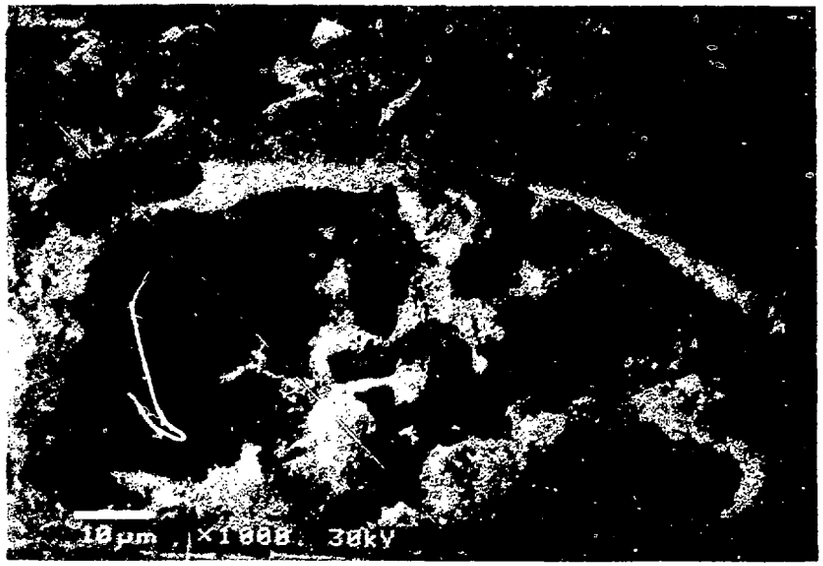


(d) Sample 29 Calcium

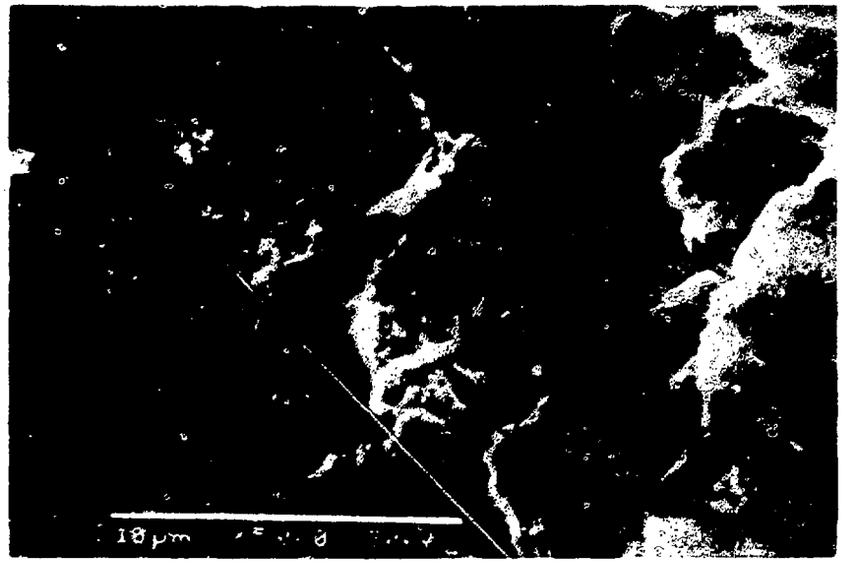
FIGURE 12. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 29, 34, 39 & 54



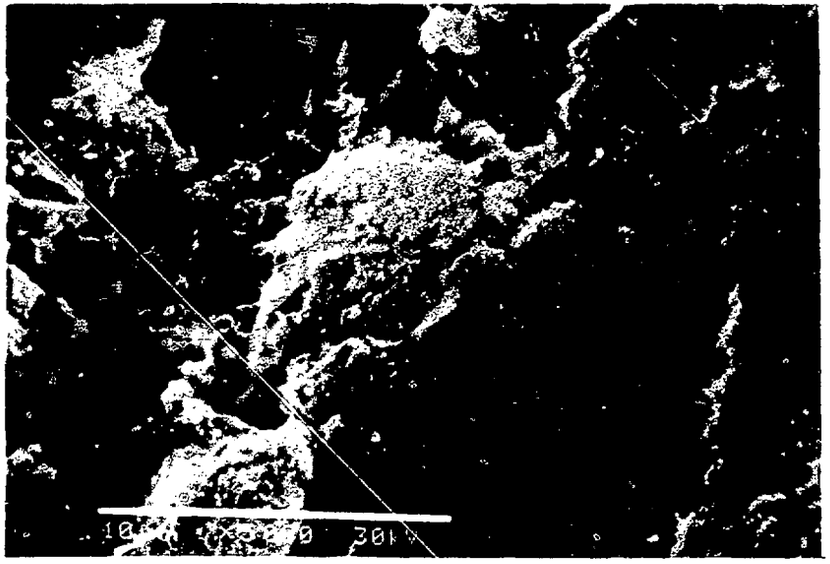
(a) Sample 7 Barium



(b) Sample 47 Lithium

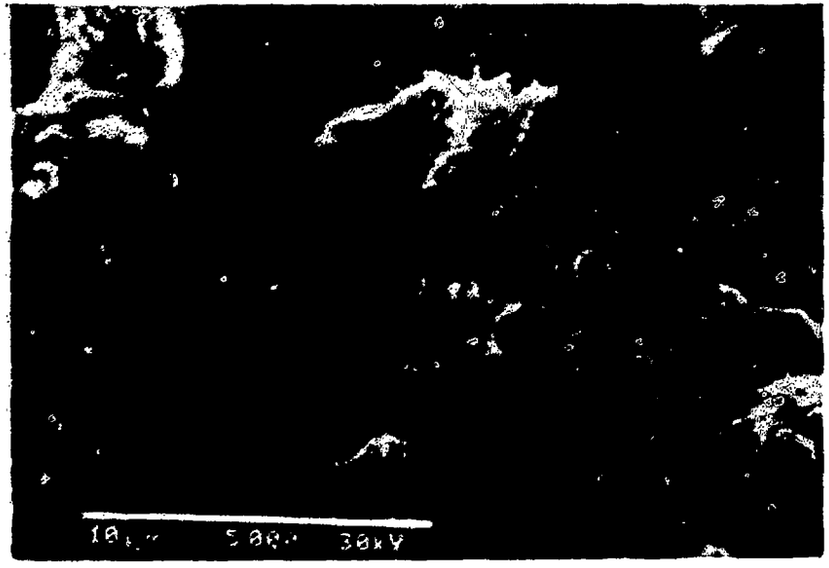


(c) Sample 15 Calcium

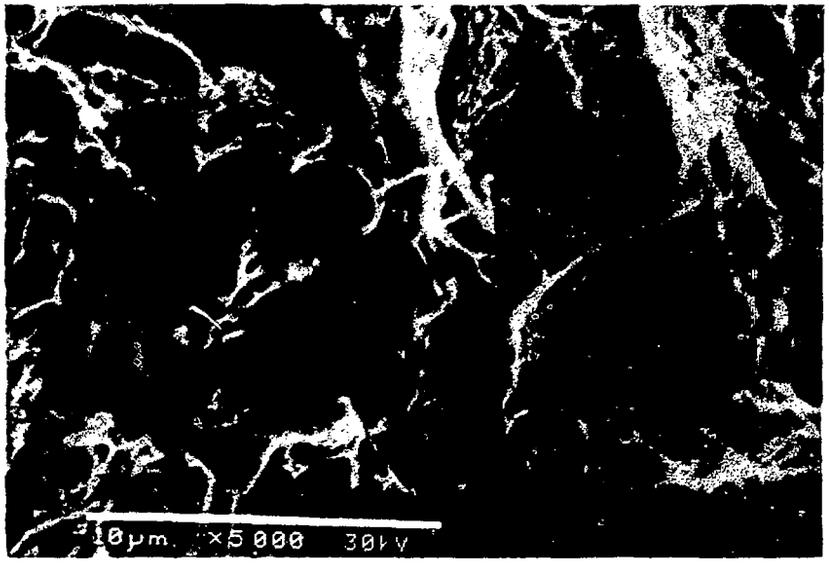


(d) Sample 64 Lithium

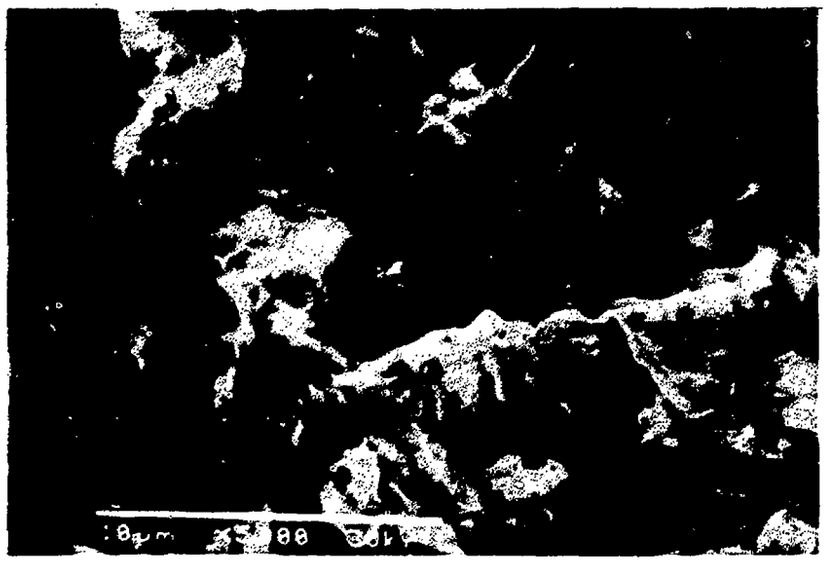
FIGURE 13. SEM MICROGRAPHS OF GREASE THICKENERS -- SAMPLES 7, 15, 47 & 64



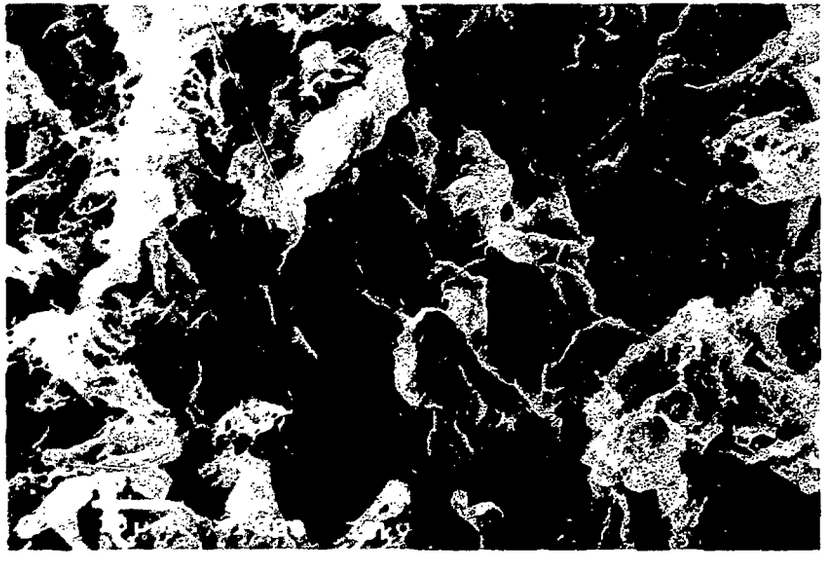
(a) Sample 26



(b) Sample 56



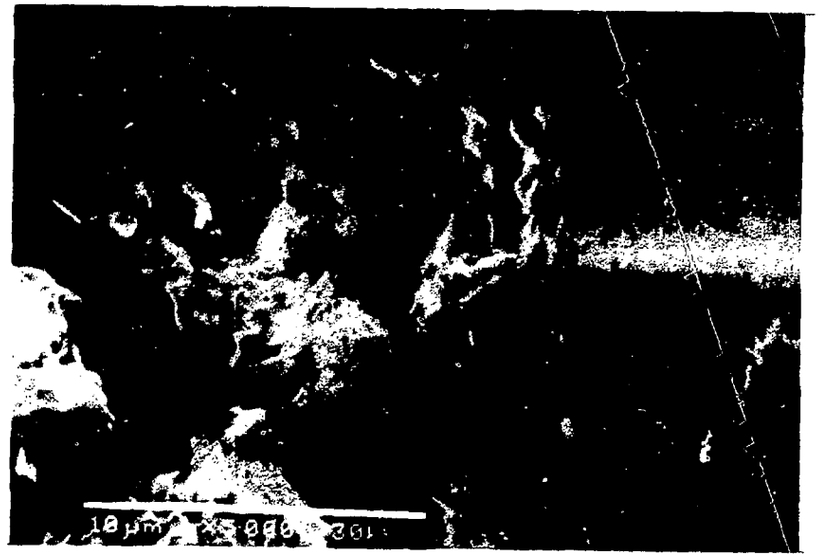
(c) Sample 27



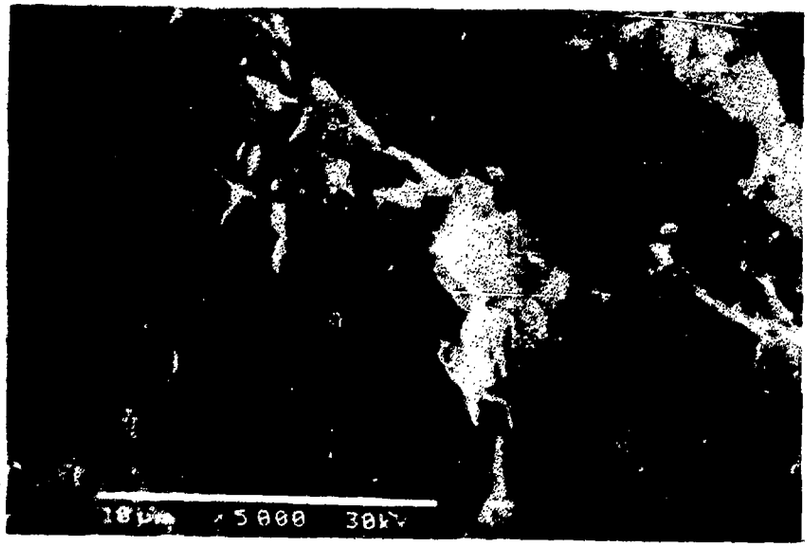
(d) Sample 9

FIGURE 14. CLAY THICKENERS -- SAMPLES 9, 26, 27 & 56

(a) Sample 25



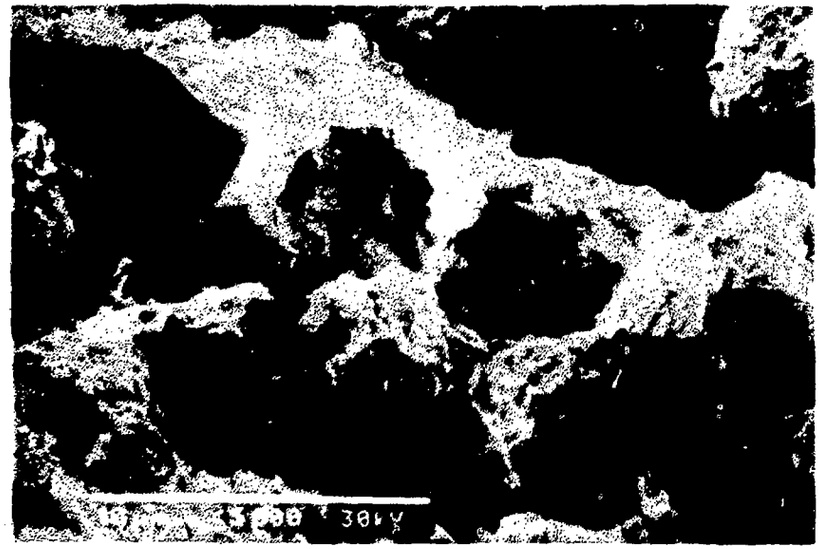
(b) Sample 38



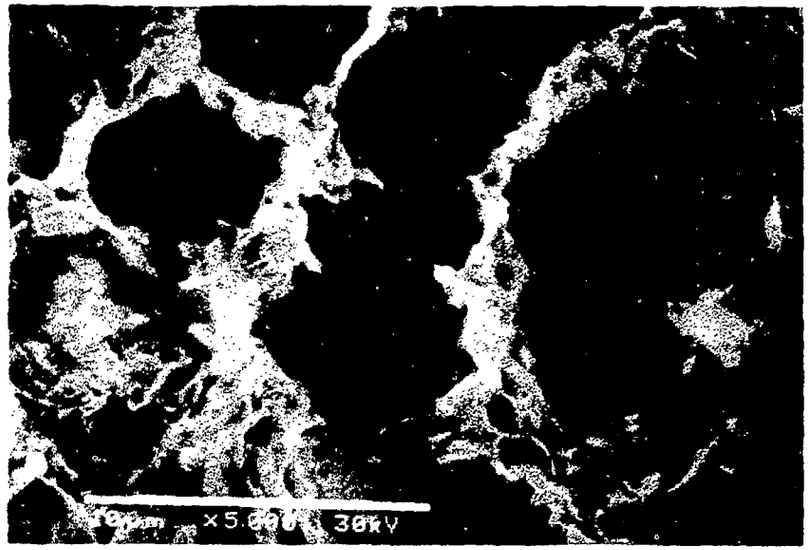
(c) Sample 24

(d) Sample 10

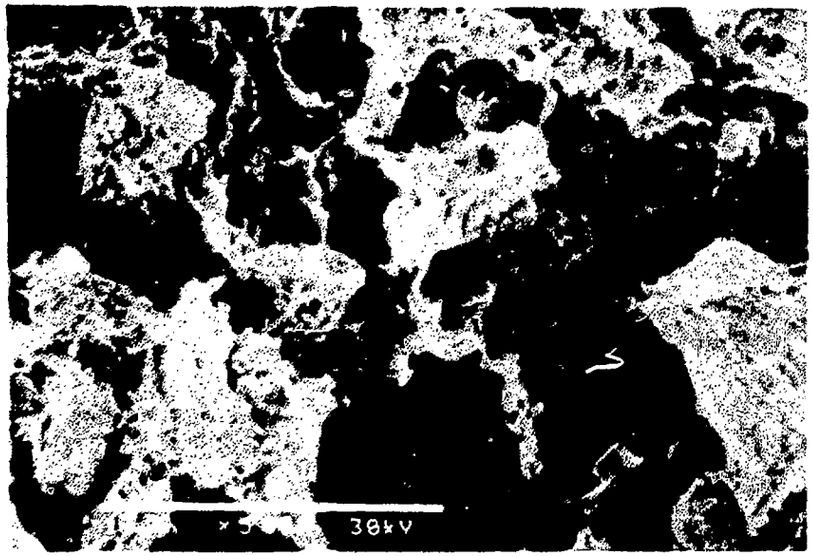
FIGURE 15. CLAY THICKENERS -- SAMPLES 10, 24, 26 & 38



(a) Sample 12



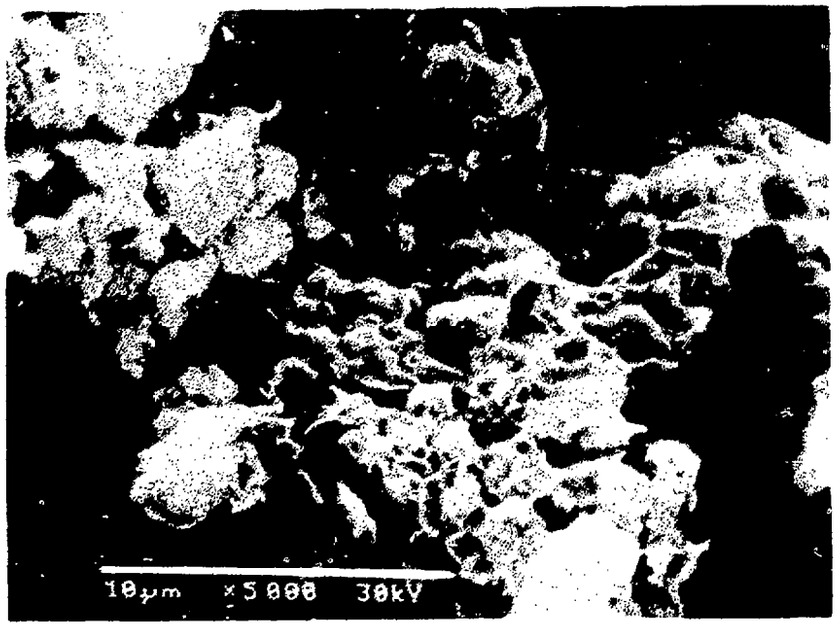
(b) Sample 41



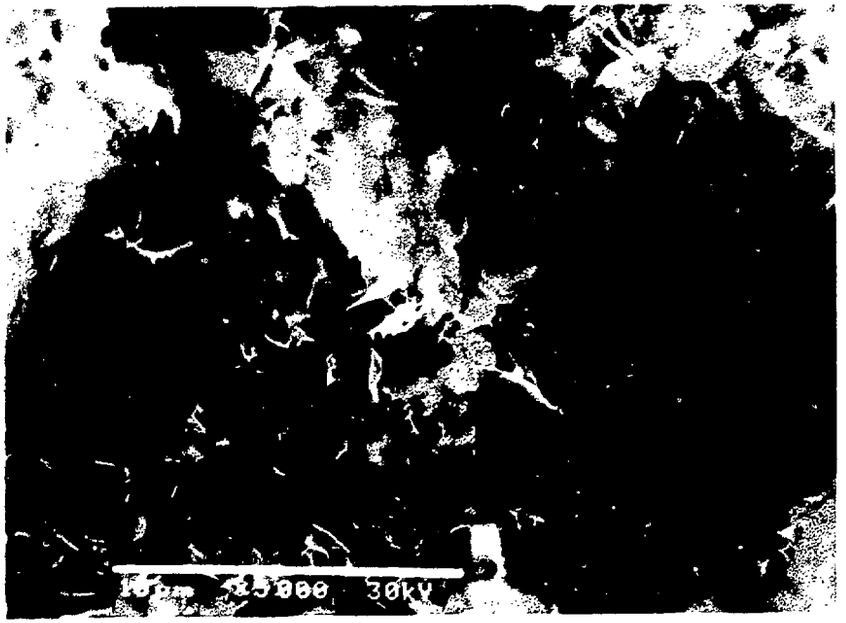
(c) Sample 11

(d) Sample 50

FIGURE 16. CLAY THICKENERS -- SAMPLES 11, 12, 41 & 50



(a) Sample 57



(b) Sample 28

FIGURE 17. CLAY THICKENERS -- SAMPLES 28 & 57

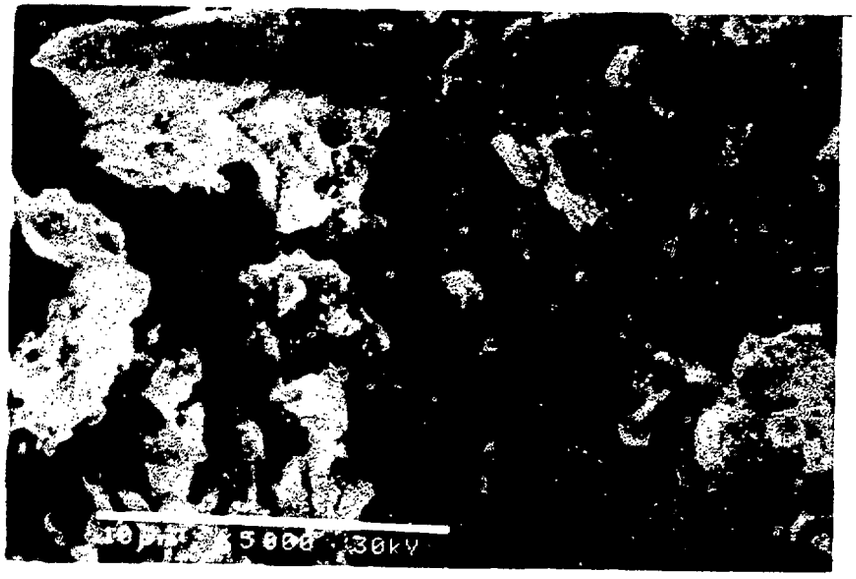
A third group of thickener microstructures are the polyurea thickeners. This group showed the widest variation of all thickener types examined. Very little similarity exists between any of the individual samples within this group. The structures for the polyurea-type thickeners are presented in Figures 18 and 19.

The fourth group examined contained only two samples, and Figure 20 represents the two microgel thickeners. It is difficult to comment on these structures since the composition of the microgel is unknown.

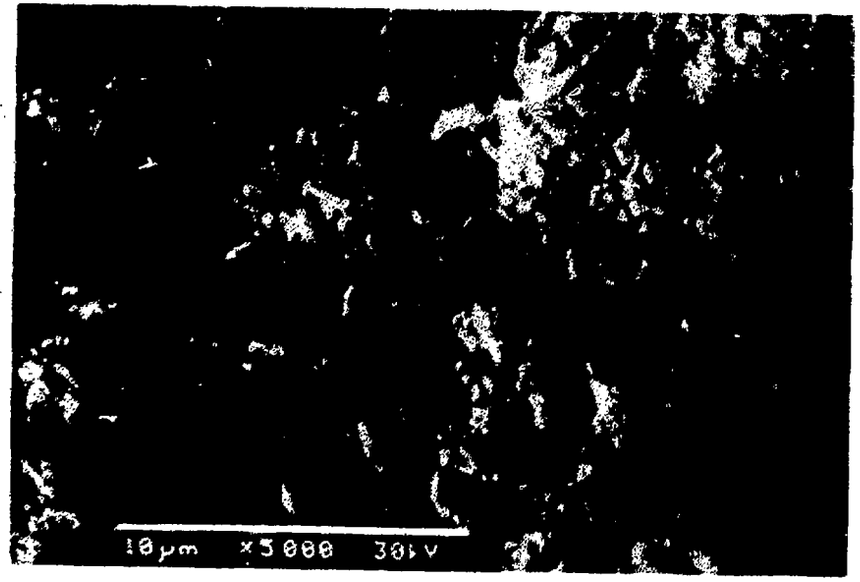
A last group of thickeners is shown in Figures 21 and 22. These photographs are included for information only since lack of thickener-type designation in Table 1 precludes identification of thickener type.

The vast variety of structures encountered within this microstructural evaluation indicates that manufacturing conditions and service history have a tremendous effect on the microstructures of the thickeners, and this may be a major factor in the performance characteristics of the greases.

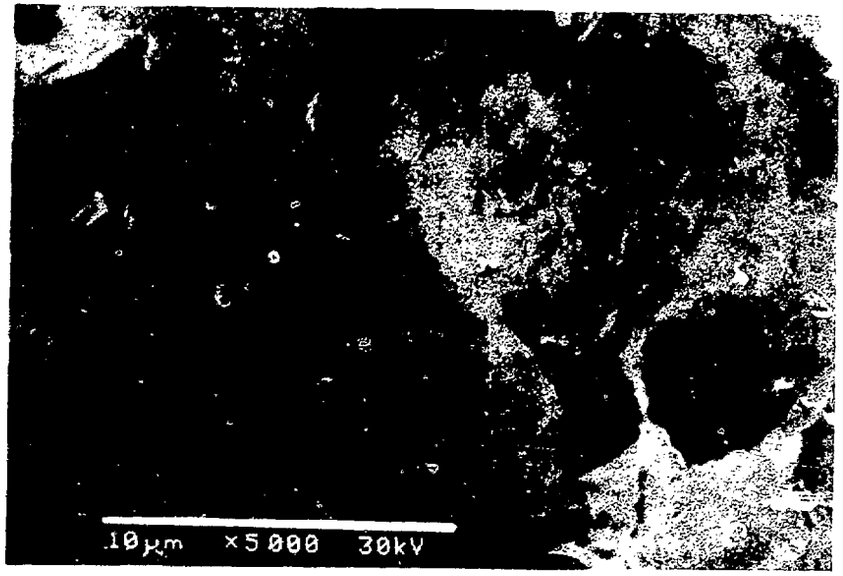
(a) Sample 1



(b) Sample 14

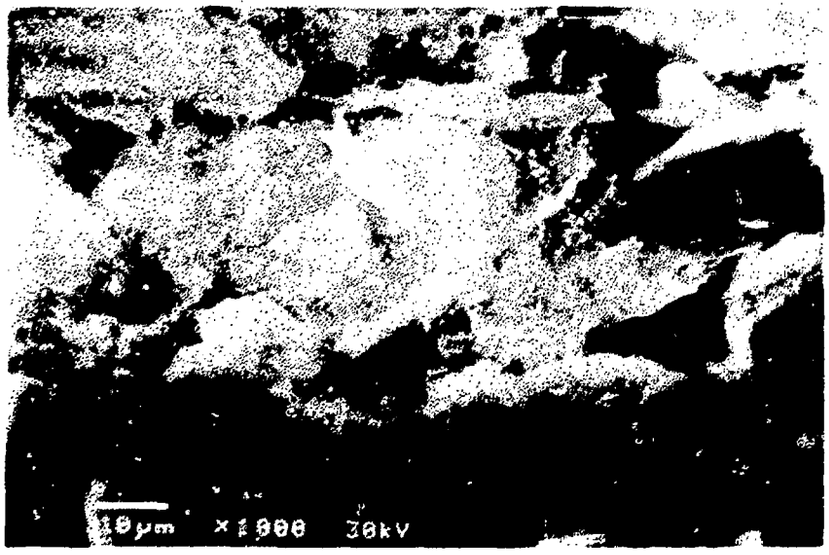


(c) Sample 61

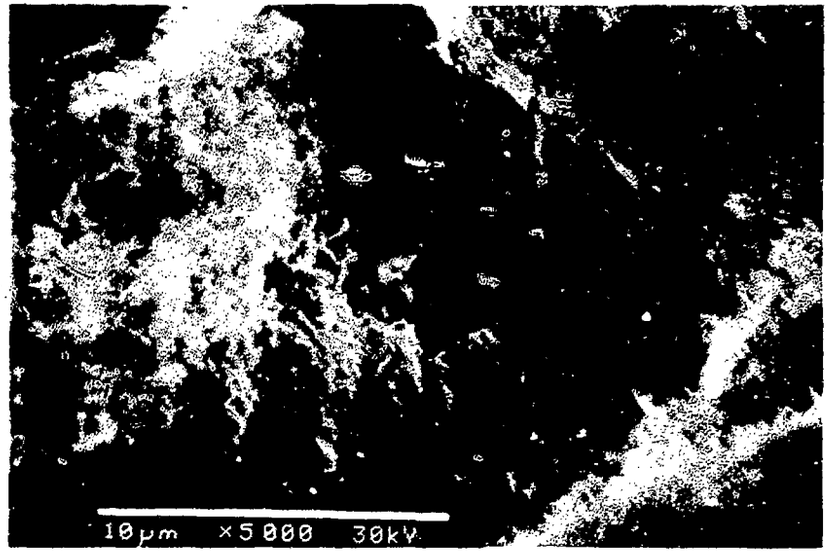


(d) Sample 45

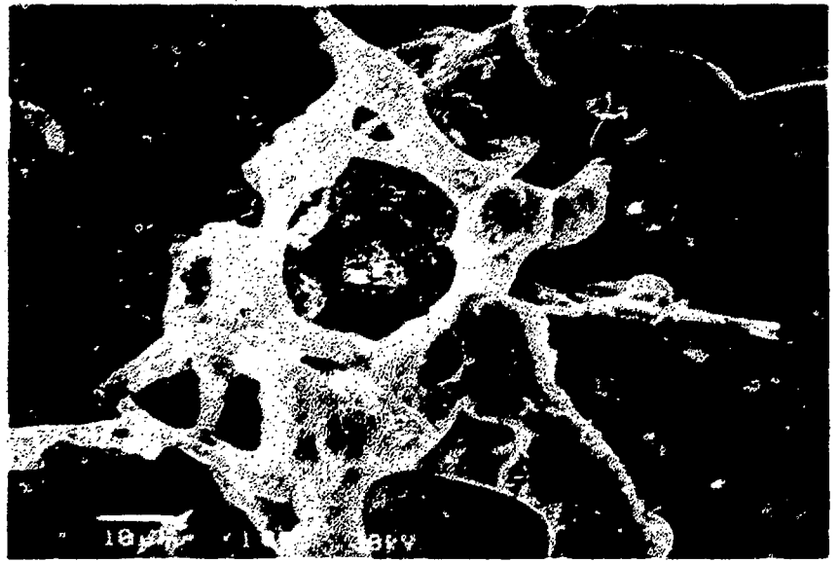
FIGURE 18. POLYUREA THICKENERS -- SAMPLES 1, 14, 45 & 61



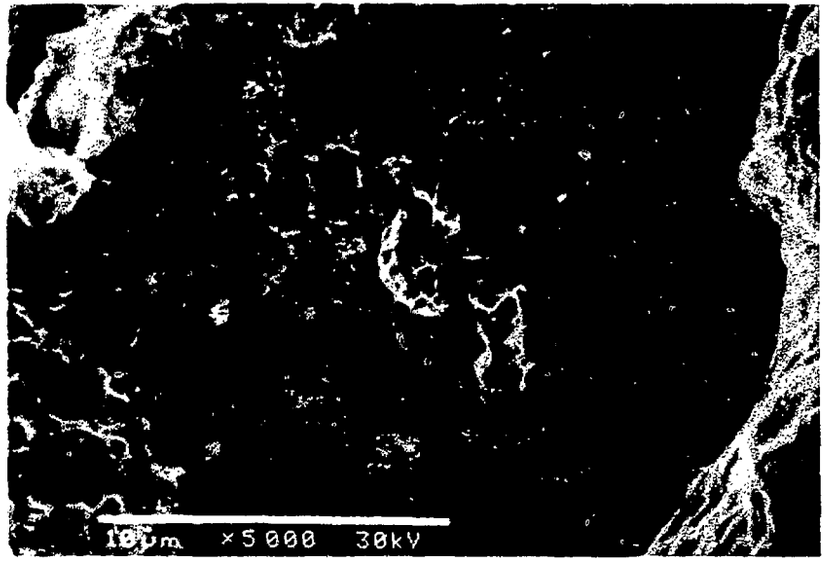
(a) Sample A66



(b) Sample A66

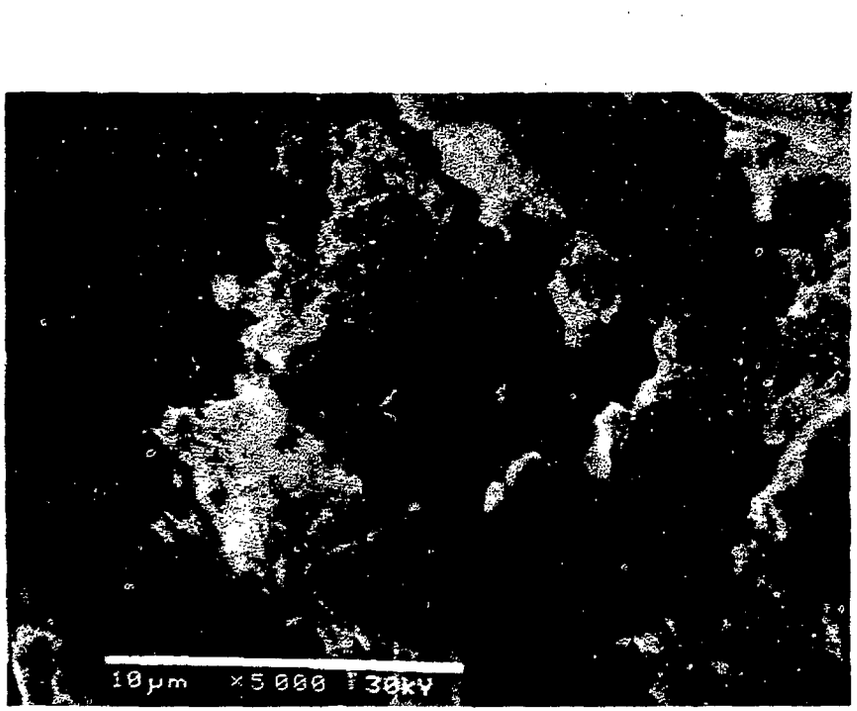


(c) Sample 30



(d) Sample 30

FIGURE 19. POLYUREA THICKENERS -- SAMPLES 30 & A66

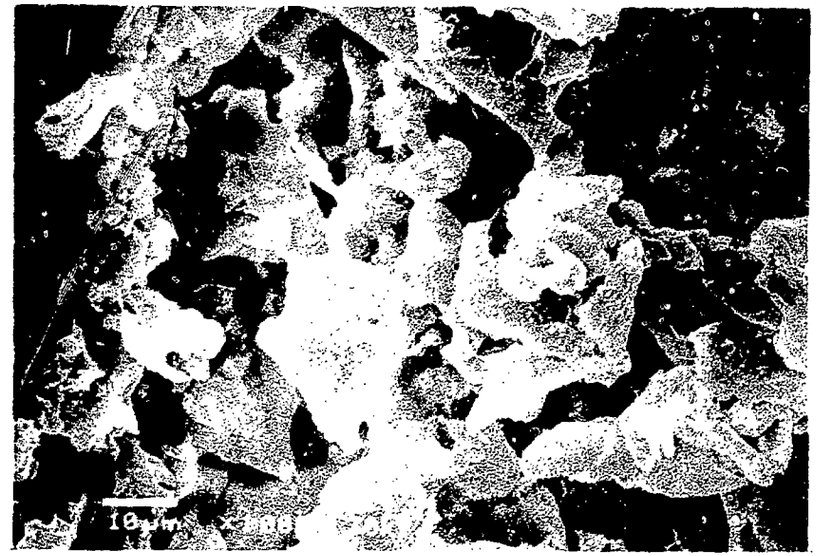


(a) Sample 18

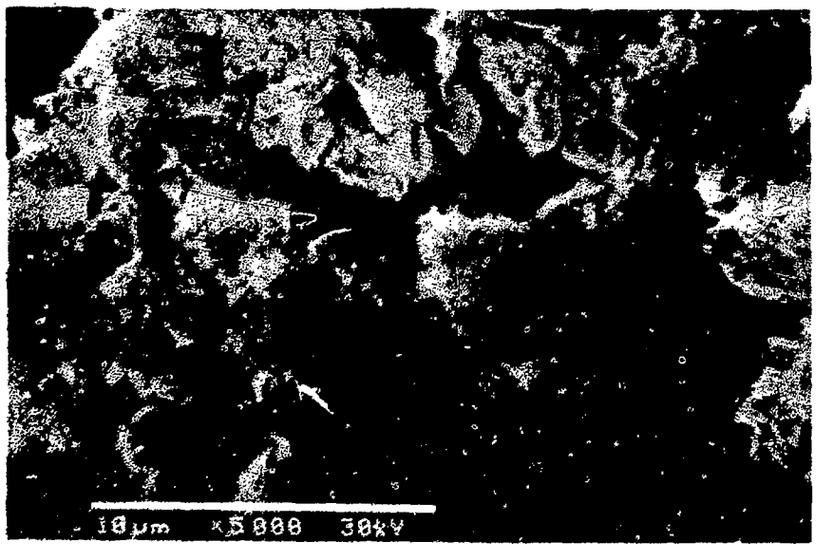
(b) Sample 40

FIGURE 20. MICROGEL THICKENERS -- SAMPLES 18 & 40

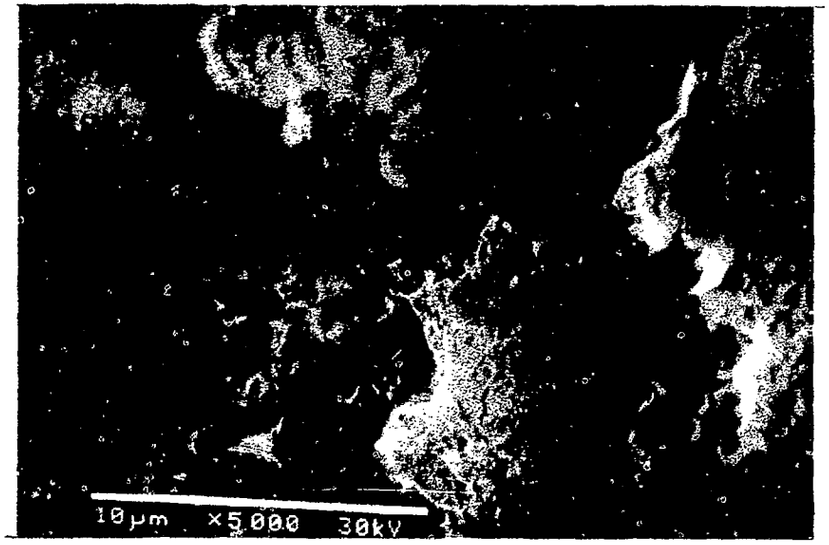
(a) Sample 36



(b) Sample 58

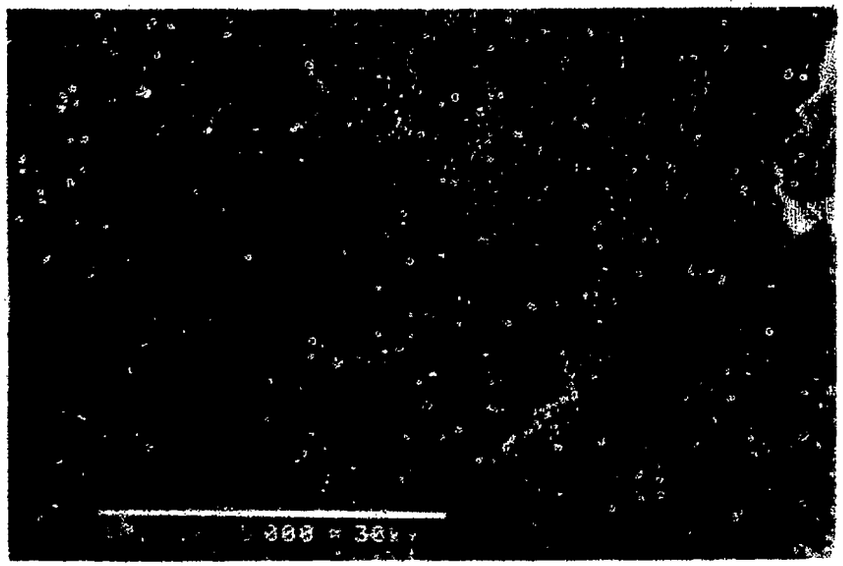


(c) Sample 43

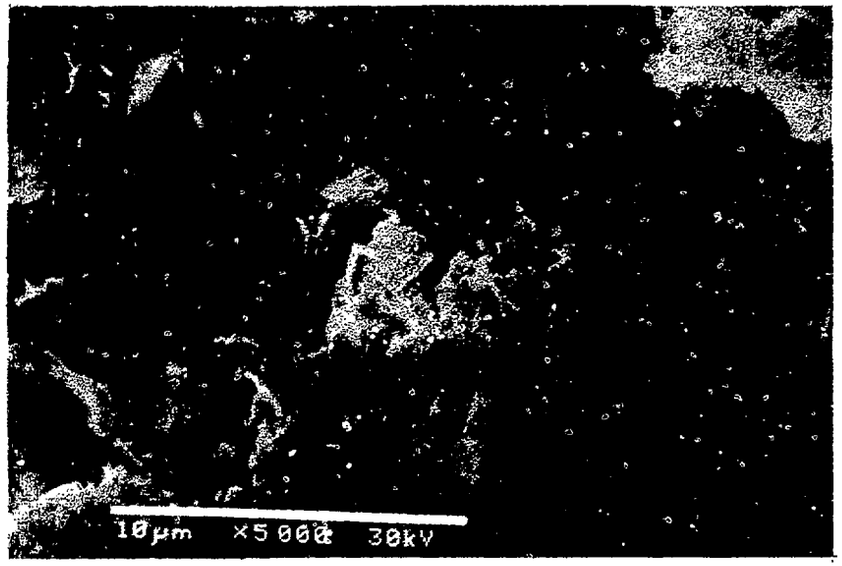


(d) Sample 31

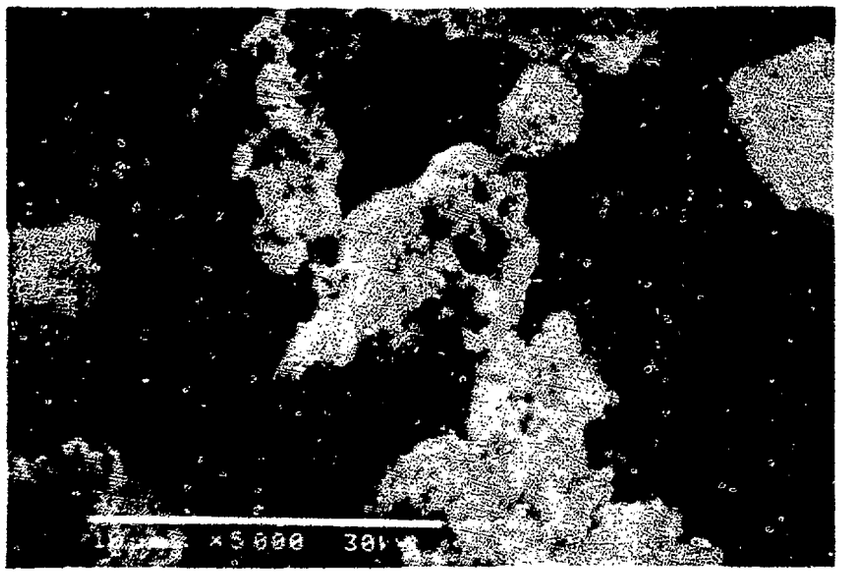
FIGURE 21. UNIDENTIFIED THICKENERS -- SAMPLES 31, 36, 43 & 58



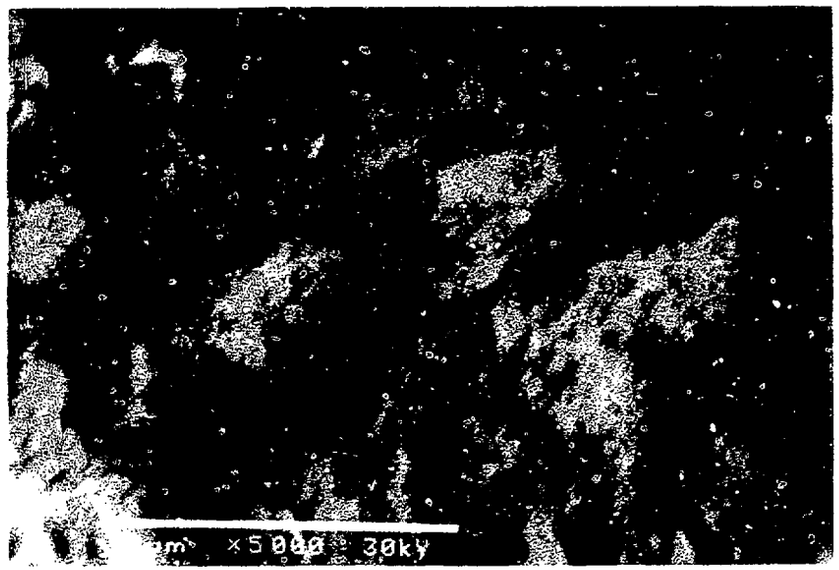
(a) Sample 32



(b) Sample 59



(c) Sample 44



(d) Sample 55

FIGURE 22. UNIDENTIFIED THICKENERS -- SAMPLES 32, 44, 55 & 59

V. CONCLUSIONS

- Solvent extraction and freeze drying of thickeners appears to be the best sample preparation approach for HPLC or SEM studies, as evidenced by the well-preserved structures, the lack of broken strands, and the ability of the thickener to reconstitute to a grease when oil is reintroduced.

- Either molecular adsorption plays a minor roll in oil/thickener interactions, or the proper conditions to allow measurement of the adsorptive qualities by HPLC were not established within the program's scope.

- The wide variety of thickener microstructures encountered in this study could account for behavioral differences between greases since the microstructures can have a tremendous effect on the thickener's ability to contain oil by mechanical entrapment or capillary effects. Examination of microstructures at several points in the service life of a grease would allow correlation between microstructural changes induced by service and overall service life.

- The apparent ability of freeze dried thickeners to reconstitute into greases could allow for the production of novel greases by using one base oil for the initial formation of an idealized microstructure, extracting this oil for reuse, and reconstituting the freeze dried thickener with an idealized base oil for the intended service. Extensive testing would be required to determine feasibility of this technique.

VI. LIST OF REFERENCES

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