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Adsorption of Surface-Active Material at the Sea/Air Interface

N. L. Jarvis

Surface Chemistry Branch
Chemistry Division

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ABSTRACT

The concentration of soluble, surface-active, organic matter in the ocean will vary with time and location. In general, sufficient surface-active material will be present to provide closely packed films at the surface under favorable conditions. The rates of adsorption of the surface-active material at a sea/air interface were studied by measuring surface tension and surface potential as a function of time. The sea water samples studied were from the ocean near La Jolla, California. Surface potential measurements indicated that adsorption of surface-active material began immediately after the interface was cleaned; however, a noticeable decrease in surface tension was not observed until the water surface was covered with a continuous film of surface-active material. Between 10 and 60 minutes were usually required to give a film having a film pressure of a few tenths of a dyne/cm, the minimum film pressure normally associated with the damping of capillary waves. Changes in surface potential and surface tension were also determined as functions of the degree of compression of the adsorbed material.

PROBLEM STATUS

This is an interim report; work on this problem is continuing.

AUTHORIZATION

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ADSORPTION OF SURFACE-ACTIVE MATERIAL AT THE SEA/AIR INTERFACE

INTRODUCTION

Most "slicks" observed on the ocean have their origin in the organic matter present in the sea. In addition to living organisms, organic constituents are often present as aggregates of nonliving material, and as soluble compounds dissolved in the sea. Recent studies have shown that the nonliving material can be a sizeable portion of the total organic content of the sea, and that much of it will be soluble in sea water (1-3). From existing data Provasoli (4) concluded that the concentration of dissolved organic matter, as measured by carbon content, is approximately 1 mg/cu liter, while from the work of Duursma (5) it is estimated that the amount of dissolved organic matter beneath a square meter of surface is of the order of 2.4 kg. Of the dissolved organic material, evidence exists (2,3,6,7) that much of it is surface active and will rapidly adsorb on bubbles present in the sea as well as at the sea surface. In a recent report, Garrett (8) identified many of the surface-active constituents present in the sea, and gave their relative abundance. Even though the amount of organic matter in the ocean will vary with location, it appears that sufficient surface-active material will be present to provide closely packed surface films under favorable conditions.

NRL has been interested in the rate at which the surface-active organic materials will adsorb at the sea/air interface, the chemical composition of the adsorbing molecules, and the effect they will have on the properties of the ocean surface. Since the rate of adsorption of the surface active molecules could not be determined directly, the rate of change of surface tension, $\Delta\gamma/\Delta t$, and of surface potential, $\Delta V/\Delta t$, were measured; these two properties of a surface are very sensitive to the presence of adsorbed molecules. Not only were changes in surface potential (ΔV) and surface tension ($\Delta\gamma$) observed with time, they were also measured as a function of the area occupied by the adsorbed molecules. By compressing the adsorbed films to smaller and smaller areas per molecule (A), information was obtained about the adsorbed molecules that is analogous to the $\Delta\gamma$ vs A and ΔV vs A studies reported in classical surface chemistry literature (9,10). These $\Delta\gamma$ vs A and ΔV vs A data not only give information as to the amount of material adsorbed, they also show the compressibility of the adsorbed film and its stability at various film pressures. In addition, they can be used to characterize the adsorbed material and are helpful in understanding the mechanisms by which these adsorbed films modify certain properties of the ocean surface. Some problems in ocean science that involve surface phenomena are the damping of capillary waves (11,12), surface temperatures (13), convective movement of surface water (14), and the stability of foams and bubbles at the sea surface.

MATERIALS AND METHODS

In view of the difficulty involved in isolating a small area of ocean surface and studying it in situ, samples of fresh sea water were quickly taken into the laboratory for prompt study. Samples were collected from the Pacific Ocean at various locations near Scripps Institution of Oceanography. Two surface samples, 0 to 10 inches deep, were collected from the end of Scripps' pier and other samples were taken at approximately two, five, and six miles west of the pier with the water depth increasing to about 1000 feet at six miles. The water temperature at the time of sampling was approximately

60°F. The sampling depth was varied from the surface to 80 feet. The subsurface water samples were collected in 16-liter Van Dorn water samplers and transported in clean 5-gallon polyethylene bottles. The water samples were taken to a laboratory at Scripps Institution of Oceanography where all experiments were carried out. In no instance did more than 45 minutes elapse from the time of collecting the samples until the beginning of the laboratory measurements. One sample of water from San Quintin Bay, Baja California, Mexico, was also studied. It was packed in dry ice during the trip to the laboratory in order to retard biological activity. The sample depth, area of sampling, and the sea surface state at the time each sample was taken are given in Table 1.

Table 1
Sea Water Samples

Sample Number	Date	Sampling Location	Sample Depth	Sea Surface Conditions
1	3 May 64	Scripps Pier	0-10"	Too rough to launch boat, surface water well mixed
2	4 May 64	2 miles west of Scripps	20'	Numerous white caps and breaking waves. Well mixed
3	4 May 64	2 miles west of Scripps	40'	Same as 2
4	8 May 64	Scripps Pier	0-10"	Occasional white caps, surface rather calm
5	7 May 64	San Quintin Bay	0-3"	Water of Bay calm but wind ruffled
6	9 May 64	6 miles west of Scripps	80'	Surface calm - gentle swells
7	9 May 64	6 miles west of Scripps	60'	Same as 6
8	11 May 64	6 miles west of Scripps	10'	Surface very calm, some slicks visible
9	11 May 64	6 miles west of Scripps	40'	Same as 8
10	12 May 64	5 miles west of Scripps	0-3"	Same as 8
11	12 May 64	5 miles west of Scripps	80'	Same as 8
12	13 May 64	6 miles west of Scripps	6'	Same as 8
13	14 May 64	6 miles west of Scripps	20'	Same as 8
14	16 May 64	6 miles west of Scripps	0-3"	Same as 8

The adsorption studies were carried out in Lucite troughs, 72.8-cm long, 12.3-cm wide, and 15-cm deep. The troughs were cleaned several times with detergent solution, rinsed thoroughly for several hours with tap water, and finally rinsed with distilled water prior to use. Rectangular glass rods were used as barriers to compress the adsorbed film and a centimeter rule was attached to the side of the tray to record the barrier position. The glass barriers and the upper edges of the Lucite tray were coated with a thin layer of paraffin to produce highly hydrophobic surfaces.

The surface tensions of the sea water samples were determined by the Wilhelmy plate method (14) using a Dognon-Abribat tensiometer (15) equipped with a platinum plate whose surface had been roughened. To insure that the plate would be completely wetted by the sea water, it was rinsed beforehand with distilled water and heated to red heat to remove any adsorbed organic material. Using this technique surface tension could be measured to within ± 0.05 dynes/cm. During each experimental run, surface tension was recorded at one minute intervals. Surface potential measurements were made by the ionizing electrode method (16,17), utilizing a 2 x 2 inch brass plate having one surface covered with thin strips of polonium 210. The alpha radiation from the polonium ionized the air gap between the electrode and the water and permitted a small current to flow between the two surfaces. The difference in potential between the water and the electrode was measured with a Keithley electrometer to a precision of ± 1 mv. The surface potential was recorded continuously on a Varian recorder. All measurements were made at room temperature, approximately $73° \pm 5°$F.

In the laboratory, the samples of sea water were placed in the Lucite film troughs and the surfaces were cleaned by sweeping with glass barriers. Usually five or six passes with the barrier were sufficient to clean the surface, i.e., there was no further increase in surface tension with subsequent sweeping. At this point the surface tension was that of clean sea water. As the initially adsorbed film was swept from the water surface, the surface potential V between the electrode and the water surface also approached a constant value. This initial surface potential was assumed to be that of a clean surface and was used as the zero or reference potential V_0. After the surface tension and surface potential values were determined for the clean surfaces, the samples were allowed to stand, and changes in surface tension ($\Delta\gamma = \gamma_0 - \gamma$) and surface potential difference ($\Delta V = V_0 - V$) were recorded as a function of time. The decreases in surface tension were plotted as changes in film pressure ($\Delta F = \gamma_0 - \gamma$). During the measurements, the film balance troughs were enclosed in clean Lucite covers to prevent contamination of the water surface by airborne particles. When the ΔF vs time and ΔV vs time curves were completed, the surface-active materials adsorbed at the interface were compressed to progressively smaller areas, and ΔF and ΔV were recorded as functions of the area occupied by the film. The adsorbed film was allowed to equilibrate for one minute after each movement of the barrier.

EXPERIMENTAL RESULTS

ΔF vs Time and ΔV vs Time

Changes in surface tension and surface potential with time were determined for twelve of the fourteen sea water samples studied. Figure 1 gives the ΔF vs time and ΔV vs time curves for the surface samples, which were collected by immersing the sampling bottle 3 to 10 inches below the water surface. Of these samples, four were collected from the Pacific Ocean near Scripps and one from San Quintin Bay, Baja California. From May 3rd to 8th when samples 1 and 4 were collected from the end of Scripps pier, the waves were from 3 to 5 feet high and many white caps were observed, indicating that the surface water was well mixed. The water was so rough the boat could not be launched from the pier. From May 9th to the end of the exercise the sea surface was calm, little or no wind was blowing, and occasional slicks were visible. Samples 16 and 14 were collected under these conditions. At San Quintin Bay there was a strong wind blowing at the time of sampling and the visible slicks were in long streaks parallel with the wind direction.

After 60 to 70 minutes in the film tray, samples 1 and 4 had higher surface potentials and film pressures than the other samples. The high values could be a result of the high sea state at the time of sampling. The high seas and breaking waves may have dispersed significant amounts of the particulate organic material present in the sea, and caused

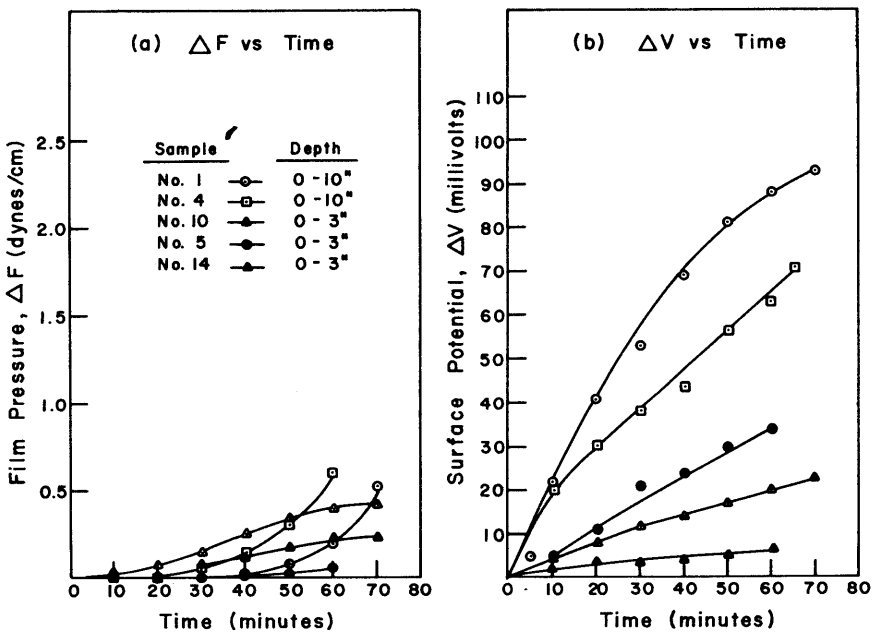

Fig. 1 - Change in film pressure (a) and surface potential (b) of a sea water surface with time as surface-active constituents adsorb at the air water interface. Sea water samples collected at sea surface.

additional material to be brought to the surface from deeper water. However, sample 10 did show a detectable film pressure much sooner than the other surface samples and gave higher film pressures during the early period of adsorption. The lowest surface potential was observed for sample 14, which was collected after the sea had been very calm for nearly a week. During this time the quantity of surface-active material in the surface layer may have been depleted by the adsorption of the surface-active material at the water surface and by the settling out of aggregates or organic matter, which Sutcliffe, Baylor, and Menzel (3) found would form when finely divided or dissolved organic material was left standing in sea water.

The data in Fig. 1 show that even in the most extreme case significant changes in surface tension were not observed for many minutes. At the end of 30 minutes, only one sample had reached a film pressure as high as 0.1 dyne/cm. Unlike surface tension, changes in surface potential were observed very shortly after the surface was cleaned. This indicates that surface-active material was adsorbing rapidly at the interface, but that several minutes were required before the surface-active material was sufficiently close packed to cause a decrease in surface tension.

Figure 2 shows the ΔF vs time and ΔV vs time curves for water samples collected at various depths. The two samples giving the highest values of ΔV were collected on May 9th, a day of relatively calm weather following a period of high seas and high winds. The subsurface samples collected on following days all gave lower values of ΔV and ΔF. This behavior is parallel to that of the surface samples where the samples collected during or shortly after rough weather show the presence of more adsorbable material. There seems to be no definite trend with depth, for sample 11 taken at 80 feet on May 12th gave ΔV and ΔF curves that are identical with those of sample 9, taken at 40 feet on May 11th. These samples are also similar to the surface samples in that they do not reach film pressures of 0.1 dynes/cm until many minutes after the surface was cleaned. In the case of sample 10 this film pressure was reached only after one hour.

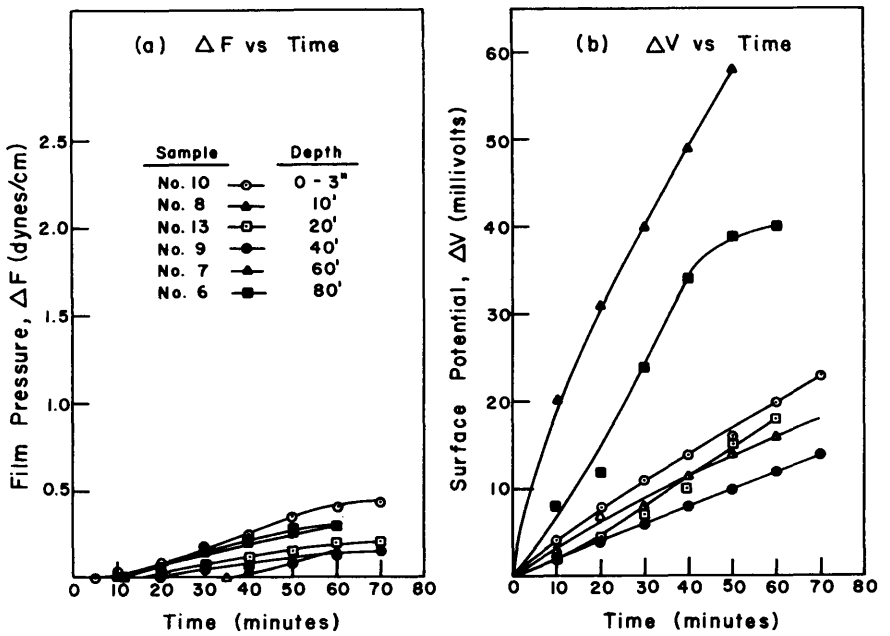

Fig. 2 - Change in film pressure (a) and surface potential (b) of a sea water surface with time as surface-active constituents adsorb at the air water interface. Sea water samples collected at various depths.

ΔF vs Area and ΔV vs Area

The sea water samples were initially allowed to stand undisturbed in the film trays while ΔV and ΔF were recorded as a function of time. The surface-active materials were allowed to adsorb into an area of approximately 800 sq cm. After a given period of time, the area occupied by the adsorbed material was gradually compressed from 800 sq cm into an area of 190 sq cm. Film pressure and surface potential measurements were made as a function of the film compression. Figure 3 gives the ΔF and ΔV values determined for films of sample 12 as they were compressed. They were determined for films of increasing age, from 15 to 207 minutes old. For films 120, 150, and 207 minutes old, the surface was not cleaned after each run, rather the film was expanded and left on the surface until the next compression. The data in Fig. 3 show that when the initial adsorption time was 30 minutes or less, very little stable film-forming material was present. Even when the area occupied by these adsorbed molecules was reduced to 190 sq cm the films did not give film pressures as high as 1.0 dyne/cm. After standing for one hour, the film developed a pressure of 4.2 dynes/cm and after 207 minutes, a film pressure of 24 dynes/cm. The increase in adsorbed surface-active material with time is also reflected in the ΔV vs area curves. The longer the surface of the sample was allowed to stand undisturbed, the greater was the measured potential of the compressed film.

The ΔF vs A curves in Fig. 3 all gave definite, measurable, film pressures at 800 sq cm and were approximately the same value. It was not until the films were compressed that differences in film composition became apparent. This suggests that the initially adsorbed films were mixtures of a number of surface-active species, varying from soluble, slightly surface-active components to highly surface-active insoluble ones. As the films were compressed, the somewhat soluble and the less stable materials were forced out of the interface. This action left the surface rich in the more surface-active

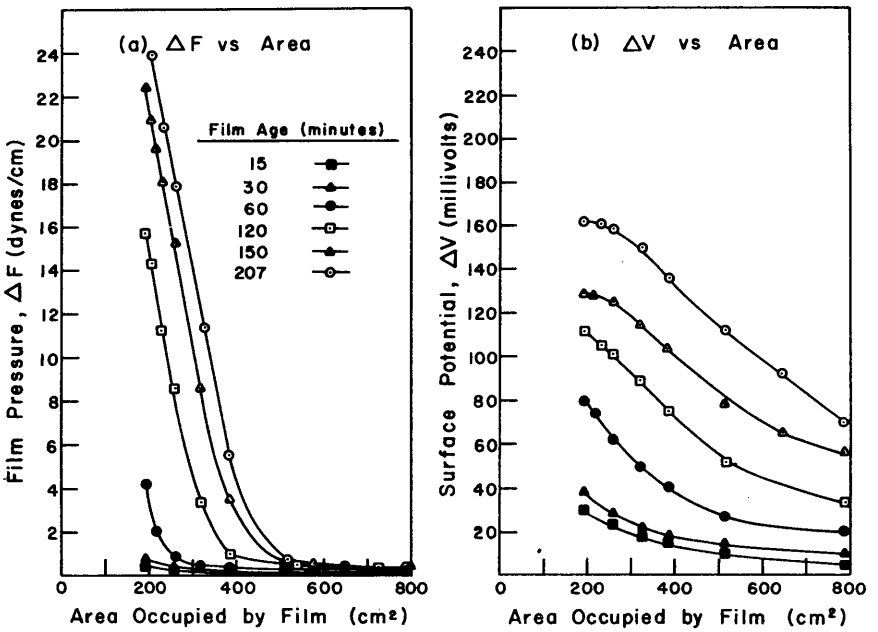

Fig. 3 - Film pressure (a) and surface potential (b) of surface-active materials as a function of film age and the area occupied by the adsorbed molecules (sample 12)

components. It was also observed that as an undisturbed surface ages the percentage of insoluble, stable film-forming material at the interface increases, indicating that the initially adsorbed soluble or slightly soluble material was gradually being replaced.

The relative rate of accumulation of the highly surface-active insoluble components will likely be increased if the film is alternately expanded and compressed. An experiment was performed with two portions of sample 5; one was allowed to stand undisturbed and the other was compressed to approximately 400 sq cm and expanded once every 5 minutes. At the end of one hour, the undisturbed sample had a film pressure of 0.2 dynes/cm while the sample that was alternately compressed and expanded had a film pressure of 0.7 dynes/cm. A curve of a single compression and expansion of one of these films also reflects the loss of material. Figure 4 is a hysteresis curve for the compression and expansion of sample 7 after aging 60 minutes. This curve is typical of each sample. The ΔF vs area expansion curves are all displaced to the left of the compression curve, are steeper, and give lower film pressures.

All samples investigated showed an increase in surface-active material at the water/air interface with time, but when the films were compressed it became apparent that all films did not contain the same amount and/or types of material. Figure 5 shows the ΔF vs area and ΔV vs area curves for several samples of near surface water that had been allowed to stand undisturbed for 60 minutes. The samples collected between May 3rd and May 8th while the sea surface was being stirred by wind and breaking waves gave much higher surface potentials and film pressures on compression. The film pressure of sample 1 began to increase when the film area was decreased from 800 to 600 sq cm, a decrease of only 25 percent. Sample 14 did not begin to show a significant increase in ΔF until the film was compressed to about 300 sq cm with the film pressure never reaching 2 dynes/cm. The ΔV vs area curves show the same behavior with the samples collected during calm weather giving the lower surface potentials.

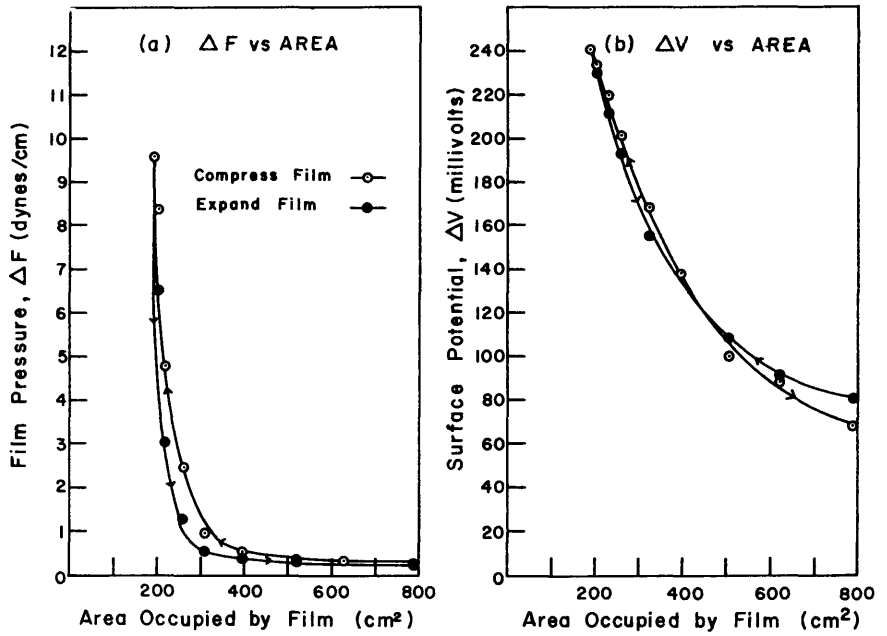

Fig. 4 - Change in film pressure (a) and surface potential (b) with compression and expansion of adsorbed monomolecular film (sample 7) (film age 60 minutes)

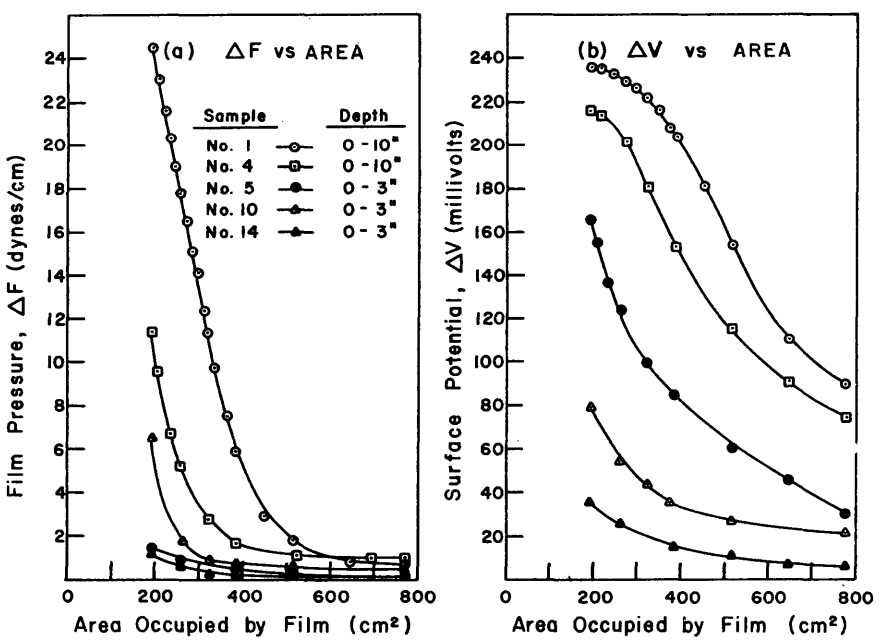

Fig. 5 - Film pressure (a) and surface potential (b) as a function of the area occupied by the adsorbed monomolecular film. Sea water samples collected from sea surface (film age 60 minutes).

The ΔF vs area and ΔV vs area curves in Fig. 6 for samples at various depths show the same general behavior, the samples collected during periods of calm weather showing much less material adsorbing at the interface with time. The one exception was sample 6, which gave a high surface potential but a low film pressure. This sample was from a depth of 80 feet, which at the time of sampling was below the thermocline. It is evident that the surfaces of these samples even after one hour are not covered with closely packed films of the insoluble surface-active material. Even sample 3, which gave the highest ΔF value, did not give a significant change in film pressure until the area had been reduced by 50 percent.

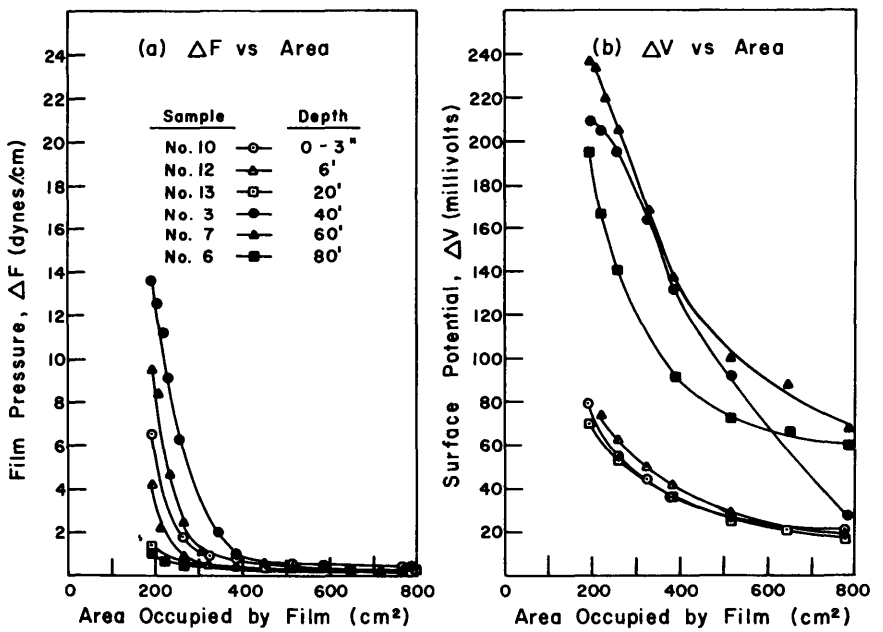

Fig. 6 - Film pressure (a) and surface potential (b) as a function of the area occupied by the adsorbed monomolecular film. Sea water samples collected at various depths (film age 60 minutes).

CONCLUSIONS

Evidence has been given that sufficient surface-active material was present in the sea water samples to form continuous monomolecular films at the water/air interfaces. Surface potential data indicated that the surface-active molecules began adsorbing at the interface immediately after the surface was cleaned. The first detectable change in film pressure, however, did not occur for several minutes until the water surface was essentially covered with a continuous film of adsorbed material. Significant differences were observed in the rate at which material was adsorbed at the surface of the samples. It was found that those samples collected when the water surface was rough and the upper water layers well mixed gave the largest changes in ΔV and ΔF.

Compression of the adsorbed films into smaller and smaller areas indicated that a portion of the initially adsorbed material was either slightly soluble or was structurally incapable of giving stable monomolecular films at higher film pressures. It was found that many of the films, even though they initially showed measurable film pressures,

failed to give a significant increase in ΔF until the area occupied by the film was reduced more than 50 percent. This could be due to the more soluble components and those structurally incapable of supporting high film pressures being forced out of the interface. It was also found that repeated compression and expansion of an adsorbed film makes it richer in the more insoluble highly surface-active components.

When a sample begins to show a film pressure of a few tenths of a dyne/cm, it is not necessarily true that the adsorbed film will be capable of damping capillary waves. Garrett and Bultman (18) have shown, from studies of insoluble films, that it is not the magnitude of ΔF that determines the damping coefficient, rather it is some function of the compressibility of the adsorbed film. They found that capillary wave damping began at the point on the ΔF vs area curve where the film pressure begins to increase rapidly with decreasing area. On this basis it is estimated that even after one hour none of the samples studied would have given films capable of damping capillary waves. In each case a certain compression of the film would have been necessary; the degree of compression required varying with the concentration of material adsorbed at each surface.

From the experimental evidence it is concluded that in those areas of the ocean comparable to the La Jolla area considerable material will accumulate at the interface within a matter of minutes in calm weather. This material will readily form "slicks" if the motion of the wind and surface water cause some compression of the adsorbed material. The extent that any film must be compressed will depend primarily upon the surface concentration or the initial state of compaction of the film.

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13. ABSTRACT

The concentration of soluble, surface-active, organic matter in the ocean will vary with time and location. In general, sufficient surface-active material will be present to provide closely packed films at the surface under favorable conditions. The rates of adsorption of the surface-active material at a sea/air interface were studied by measuring surface tension and surface potential as a function of time. The sea water samples studied were from the ocean near La Jolla, California. Surface potential measurements indicated that adsorption of surface-active material began immediately after the interface was cleaned; however, a noticeable decrease in surface tension was not observed until the water surface was covered with a continuous film of surface-active material. Between 10 and 60 minutes were usually required to give a film having a film pressure of a few tenths of a dyne/cm, the minimum film pressure normally associated with the damping of capillary waves. Changes in surface potential and surface tension were also determined as functions of the degree of compression of the adsorbed material.

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14. KEY WORDS: Key words are technically meaningful terms or short phrases that characterize a report and may be used as index entries for cataloging the report. Key words must be selected so that no security classification is required. Identifiers, such as equipment model designation, trade name, military project code name, geographic location, may be used as key words but will be followed by an indication of technical context. The assignment of links, roles, and weights is optional.