



Nonionic Wetting Agents

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ABSTRACT

Purified polyglycol ester and ether types of nonionic surfactants were investigated to determine optimum structures for wetting hydrophobic soils, peat moss, and standard cotton skeins (Draves test). The most rapid wetting of hydrophobic soil was observed with diethylene glycol monoesters of fatty acids possessing 8,9, and 10 carbon atoms and triethylene glycol monoesters of fatty acids containing 9 and 10 carbon atoms. Methyl pentaethylene glycol decanoate and methyl hexaethylene glycol dodecanoate were also effective wetting agents for hydrophobic soil. Nonionics with slightly longer oxyalkylene chains most effectively wet Draves skeins and peat moss. Diethylene glycol and triethylene glycol monodecyl ethers also effectively wet hydrophobic soil and peat moss.

INTRODUCTION

Chemical structural requirements for efficient anionic wetting agents have been studied by a number of workers (1,2). It has been shown that the best anionic wetting agents have their hydrophilic group located near the center of the molecule, while anionic surfactants having the hydrophilic functional group located near one end of the hydrophobic chain function primarily as detergents, lime soap dispersing agents, and emulsifiers. Other structural elements which favor higher critical micelle concentration, such as shorter hydrophobic alkyl chains, also enhance wetting properties.

A search of the literature did not reveal analogous studies relating chemical structure of nonionic surfactants to wetting properties. This information would be particularly helpful in the design of suitable surfactants for the treatment of hydrophobic soil conditions (3). Such conditions are encountered after forest fires, in groves of older citrus trees, and the production of ornamental plants in peat moss. In general, nonionic wetting agents would be useful in such applications since hard water ions might inhibit the effectiveness of anionic wetting agents. Up to the present, studies on the treatment of hydrophobic soils have been limited to commercially available nonionic surfactants. Unfortunately, such commercial materials were generally designed to function as detergents and emulsifiers and may not possess optimum wetting properties. Furthermore, commercial nonionic surfactants, because of the nature of the oxyethylation process, are not pure compounds but mixtures of compounds whose degree of oxyethylation is spread over a broad range, usually following a typical Gaussian distribution curve.

In the present study, highly purified nonionic surfactants were prepared and examined for wetting properties. The structure of nonionic surfactants can be modified conveniently by: a) varying the size and structure of the hydrophobic (hydrocarbon) portion, b) changing the size of the hydrophilic (polyoxyethylene) portion of the molecule, or c) altering the position of the hydrophilic group or

groups on the hydrophobic chain, in analogy to what has been done with anionic surfactants. In order to study the effects of alkyl chain length, oxyalkylene chain length, and the balance between them, fatty acids of 8 to 14 carbon atoms were esterified with various purified homogeneous (monodisperse) polyethylene glycol fractions having 2 to 6 oxyethylene groups. On the basis of the structure of those polyglycol esters possessing optimum wetting properties, analogous purified fatty acid esters of monomethyl ethers of polyglycols were prepared. Homogeneous ether alcohols having a hydrophilic and hydrophobic content similar to those of the above were also synthesized and evaluated as wetting agents for hydrophobic soil, peat moss, and treated cotton skeins.

EXPERIMENTAL PROCEDURES

Materials

Octanoic, nonanoic, decanoic, dodecanoic, tetradecanoic, hexadecanoic, and 10-undecenoic acids and octanol, decanol, and dodecanol were purified from commercial materials by standard distillation and crystallization procedures. Each of these materials was found to have purity in excess of 98% by gas liquid chromatography (GLC).

Polyethylene glycols and their monomethyl ethers from commercial sources or laboratory preparations were carefully fractionated by vacuum distillation through a column 1¼ in. diameter by 12 in. long, packed with Cannon stainless steel protruded packing (¼ in. by ¼ in.), (Applied Science Laboratories, Inc., State College, PA). Purity of the fractions determined by GLC analysis is shown in Table I.

Synthetic Procedures

Esterifications. A six to ten molar excess of polyethylene glycols was used in the preparation of monoesters in order to minimize diester formation. The synthesis of diethylene glycol monodecanoate is illustrative of the

TABLE I
GLC Analysis of Polyethylene Glycol Fractions

	Purity, %	Principal impurity
H(O ₂ H ₄) ₂ OH	96	3% i = 1 ^a
H(O ₂ H ₄) ₃ OH	98	2% i = 2
H(O ₂ H ₄) ₄ OH	99	—
H(O ₂ H ₄) ₅ OH	92	6.5% i = 6
H(O ₂ H ₄) ₆ OH	99	—
CH ₃ (O ₂ H ₄) ₃ OH	92	4% i = 2, 2% i = 4
CH ₃ (O ₂ H ₄) ₄ OH	98	2% i = 3
CH ₃ (O ₂ H ₄) ₅ OH	85	13% i = 4
CH ₃ (O ₂ H ₄) ₆ OH	99	—
CH ₃ (O ₂ H ₄) ₇ OH	94	6% i = 8
CH ₃ (O ₂ H ₄) ₈ OH	91	8% i = 7

^ai = number of oxyethylene groups.

TABLE II

Wetting Properties of 0.1% Aqueous Solutions of Polyethylene Glycol Monoesters
 $C_nH_{2n+1}CO_2(C_2H_4O)_iH$

No.	n	i	HLB ^a	Solution appearance	Purity ^b %	Draves test ^c sec	Glendora soil ^d	
							Wet, sec	Rewet sec
1	7	2	7.8	Separation	92	>300	1	0 ^e
2	7	3	9.6	Cloudy	78	>300	180	35
3	7	4	11.0	Cloudy	92	>300	535	44
4	8	2	7.2	Separation	90	72	0 ^e	0 ^e ‡
5	8	3	9.1	Cloudy	90	6	15	0 ^e
6	8	4	10.6	Cloudy	90	48	102	20
7	8	5	11.7	Clear	89	95	670	70
8	9	2	6.8	Separation	87	41	1	4
9	9	3	8.7	Cloudy	87	7	12	0 ^e
10	9	4	10.1	Cloudy	92	11	11	15
11	9	5	11.2	Clear	97	12	507	36
12	9	6	12.1	Clear	92	— ^f	1087	30
13	11	3	8.0	Separation	82	>300	600	100
14	11	4	9.4	Cloudy	90	23	75	0 ^e
15	11	5	10.5	Cloudy	87	7	5	3
16	11	6	11.4	Clear	86	34	193	20
17	13	5	9.8	Cloudy	84	52	240	2
18	13	6	10.7	Cloudy	80	21	260	15
19	10 [§]	3	8.3	Cloudy	93	9	210	45
20	10 [§]	4	9.7	Cloudy	94	6	300	5
21	10 [§]	5	10.8	Cloudy	93	7	210	45

^aHydrophilic = lipophilic balance calculated by Griffin's formula (11).

^bPurity calculated from hydroxyl values.

^cTime required for a standard cotton skein to sink (8).

^dDrop penetration time (7). Rewet with distilled water.

^eImmediate wetting, too fast to measure.

^fInsufficient sample.

[§]Undecenoic acid.

method of preparation: 4.4 g (0.0255 mole) of decanoic acid, 19.3 g (0.214 mole) of diethylene glycol, and 0.18 g of p-toluenesulfonic acid were heated in 60 ml of refluxing toluene for 4 hr, and 1.5 ml of water was removed azeotropically. After cooling, catalyst and excess glycol were removed by washing three times with demineralized water. Toluene and other solvents were removed in a vacuum rotary evaporator, leaving 6.1 g of an oily residue. Hydroxyl content and hence monoester content was determined by acetylation and titration (4). Monoester content was also determined directly by reverse phase high performance liquid chromatography (HPLC) with an 80:20 methanol/water mixture as solvent (5). The free fatty acid content of the esters was determined by titration. Monoester contents of the test esters determined from hydroxyl values are listed along with wetting properties in Table II.

Diesters were prepared by refluxing 0.55 mole of the appropriate polyethylene glycol per mole of fatty acid in toluene for 24 hr with an acid catalyst and isolating the product in the usual manner. Properties of diester and their mixtures with corresponding monoesters are listed in Table III.

Since monomethyl ethers do not have a second hydroxyl group and consequently cannot form diesters, only a 15-20% excess of glycol ether was used in the preparation of their esters. Purity was determined by standard saponification number methodology. Properties are listed in Table IV. All compounds gave saponification numbers within 6% of the expected theoretical values.

Ether alcohols. Octanol, decanol, and dodecanol were reacted with ethylene oxide in a conventional manner (6) to give a mixture of randomly distributed ether alcohols. The mixtures were separated by vacuum distillation through a column, 1¼ in. diameter x 12 in. long, packed with Cannon protruded packing and analyzed by GLC and TLC. Purity and wetting properties are shown in Table V.

Evaluations were carried out with both the separated homogeneous compounds and reaction mixtures where average numbers of ethylene oxide units are shown in parentheses. The purity of the ether alcohols of this study was determined by hydroxyl value and confirmed by gas liquid chromatography; the degree of purity of each ether alcohol sample is shown in Table V.

Evaluation

Hydrophobic soils. Two samples of burned-over forest soils were kindly supplied by N. Valoras of the University of California at Riverside. They were Glendora, a sandy soil taken from the Angeles National Forest near Glendora, CA, and Idylwild, a shale soil from the San Jacinto mountain area near Idylwild, CA. Larger particles were removed from Glendora soil by passing it through a twenty mesh screen. A hydrophobic soil sample taken under an old citrus tree near Dundee, FL, was kindly supplied by Dr. Robert Koo, Agricultural Research and Education Center, Lake Alfred, FL. All soil samples were air-dried and stored in air-tight containers to prevent changes in moisture content while the tests were in progress.

Canadian sphagnum peat moss containing 24% moisture was ground in a Wiley mill with a No. 10 screen and stored in an air tight container without further drying.

Evaluation procedures. The drop penetration time (DPT) employed by Letey and coworkers (7) was adapted as a screening test for surfactants. One gram of soil or ground peat moss was given a smooth slightly concave surface with a spoon-shaped spatula. A 0.1 ml drop of 0.1% aqueous solution of the test material was applied to the surface, and time was recorded for the complete disappearance of free water. Rewetting was measured by placing a 0.1 ml drop of distilled water on the surfactant-treated substrate which had dried by standing overnight at room temperature and a relative humidity of 20-50%.

TABLE III
Wetting Properties of Mixtures of Monoesters and Diesters
of Diethylene Glycol and Octanoic Acid (0.1%)

No.	Composition		Glendora DPT ^a		Peat moss DPT ^a	
	% Monoester	% Diester	Wet, sec	Rewet, sec	Wet, sec	Rewet, sec
1	92 ^b	8	0	3	9	7
2	89 ^c	11	2	0	75	600
3	73 ^d	27	8	14	100	660
4	70 ^d	30	25	11	770	900
5	63 ^d	37	240	0	600	1800
6	39 ^d	61	900	12	>5000	>5000
7	3 ^c	97	660	192	>5000	>5000

^aDrop penetration time (7).

^bBased on hydroxyl value.

^cAnalyzed by HPLC.

^dCalculated from components of mixture.

TABLE IV
Wetting Properties of 0.1% Aqueous Solutions of Methyl Polyethylene Glycol Esters
 $C_nH_{2n+1}CO_2(C_2H_4O)_iCH_3$

No.	n	i	Solution appearance	Draves test ^a sec	Glendora soil ^b		Peat moss ^b	
					Wet, sec	Rewet, sec	Wet, sec	Rewet, sec
1	7	4	Cloudy	>300	840	4	>5000	>5000
2	7	5	Clear	>300	1020	15	>5000	>5000
3	8	3	Separation	>300	615	40	330	>5000
4	8	4	Cloudy	248	690	100	4440	420
5	8	5	Cloudy	12	960	5	>5000	1920
6	8	6	Clear	23	420	100	1290	270
7	9	3	Separation	34	610	90	>5000	540
8	9	4	Cloudy	24	180	18	>5000	>5000
9	9	5	Cloudy	9	8	31	2	120
10	9	6	Cloudy	-- ^c	390	46	10	21
11	9	7	Clear	9	1030	145	65	450
12	11	4	Separation	288	105	25	11	180
13	11	5	Cloudy	12	44	22	33	115
14	11	6	Clear	5	9	15	12	180
15	11	7	Clear	9	80	0	105	70
16	11	8	Clear	10	35	3	110	180
17	13	5	Cloudy	64	540	0	600	60
18	13	6	Clear	35	295	5	450	60
19	13	7	Clear	17	480	36	75	70
20	15	6	Curd	>300	750	30	1260	165
21	15	7	Cloudy	>300	360	31	1800	60

^aTime required for a standard cotton skein to sink (8).

^bDrop penetration time (7). Rewet with distilled water.

^cInsufficient sample.

The modified Draves wetting test (8) was used to measure fabric wetting ability at room temperature. In order to screen a large number of compounds, measurements were limited arbitrarily to 0.1%. Wetting properties are summarized in Tables II, III, IV, and V.

RESULTS AND DISCUSSION

Synthesis

Since the objective of this study was to prepare nonionic surfactants of high purity and determine their wetting characteristics, fatty acid monoesters were synthesized from individual polyglycols of 85-99% purity (Table I). The principal impurities of the purified polyglycols were polyglycols containing one oxyethylene group more or less than the main fraction.

The formation of monoesters was always accompanied by diester formation, and there was some indication that monoesters upon standing rearranged to form more diester (9). Because of the diester content of the monoester preparations and contamination by residual solvent, the assessment of purity of the monoesters by saponification number proved unreliable. Calculation of purity from

hydroxyl values gave more reliable data which could be confirmed by direct determination of mono- and diester content by HPLC. Agreement between these two analytical methods was within 2%. The monoester content of the preparations varied from 78 to 97% as shown in Table II. It has been stated in the literature that diester byproducts are always present in monoester preparations regardless of whether the latter are synthesized via direct oxyethylation or by esterification of fatty acid with a polyglycol (10).

Evaluation Methodology

We anticipated in the initial phases of this study that contact angle measurement on hydrophobic surfaces such as Teflon, polyethylene, or polypropylene would give reliable, reproducible data that would correlate well with practical application wetting tests discussed below. Unfortunately, the angle changed so rapidly that it was difficult to obtain good reproducible contact angle data, and no correlation between contact angle measurements and wetting tests could be seen; hence, contact angle data are not reported here. The wetting tests consist of the cotton skein sinking test (8), modified Draves test, commonly used in the textile field, and drop penetration tests on three

TABLE V
Wetting Properties of 0.1% Aqueous Solutions of Ether Alcohols
 $C_nH_{2n+1}(OC_2H_4)_iOH$

No.	n	i(a) ^a	HLB ^b	Solution appearance	Purity %	Draves test ^c sec	Glendora soil ^d		Peat Moss ^d	
							Wet, sec	Rewet, sec	Wet, sec	Rewet, sec
1	8	1	5.1	Separation	75 ^e	>300	135	48	>5000	4800
2	8	2	8.1	Separation	98 ^e	5	300	4	>5000	>5000
3	8	3	10.1	Clear	90 ^f	22	345	180	>5000	>5000
4	8	4	11.5	Clear	70 ^f	60	495	120	>5000	2160
5	8	(2.6)	9.4	Clear	—	9	106	20	3600	>5000
6	10	1	4.4	Separation	96 ^e	>300	11	300	>5000	>5000
7	10	2	7.1	Separation	96 ^e	10	0	15	0	270
8	10	3	9.1	Cloudy	80 ^f	4	0	10	8	285
9	10	4	10.5	Clear	70 ^f	5	10	15	20	540
10	10	(3.2)	9.4	Cloudy	—	50	5	0	— ^g	— ^g
11	12	3	8.3	Cloudy	98 ^f	39	160	0	60	180
12	12	4	9.7	Cloudy	98 ^f	46	650	80	135	525
13	12	(5.1)	10.9	Cloudy	—	14	45	17	25	360
14	12	(8.1)	13.1	Clear	—	11	450	64	7	180
15	12	(9.6)	14.0	Clear	—	11	720	134	33	225

^ai = Number of oxyethylene groups of pure individuals. Numbers in parentheses are averages of reaction mixtures determined by weight increase on oxyethylation.

^bHydrophilic-lipophilic balance calculated by Griffin's formula (11).

^cTime required for a standard cotton skein to sink (8).

^dDrop penetration time (7). Rewet with distilled water.

^ePurity by GLC; impurities are isologs with different values of i.

^fEstimation based on TLC and hydroxyl values.

^gInsufficient sample.

hydrophobic soils and ground peat moss.

Most wetting tests, such as the Draves test, involve the determination of the concentration of surfactant required to wet the substrate in a fixed time interval, e.g., 25 sec for the Draves test. This becomes a time-consuming task, and for practical reasons we decided to run all wetting tests at a fixed surfactant concentration of 0.1% of sample regardless of purity. All wetting and rewet times of 60 sec or less are reproducible within $\pm 5\%$.

In any treatment of hydrophobic soil with wetting agent, both wetting time, i.e., the time required for the surfactant treatment to penetrate the hydrophobic soil, and rewet time, i.e., the time required for rain or irrigation water to penetrate the surfactant-treated soil, are of equal importance. Accordingly, data for both were recorded in the tables. A comparison of drop penetration tests on Glendora, Idylwild, and Florida citrus orchard soil revealed that Glendora soil was substantially more hydrophobic than the other two soils, i.e., those surfactants capable of wetting Glendora soil consistently wet the other two soils even more rapidly. Thus, Glendora soil test data appeared to be the most meaningful and are recorded here in the tables below.

Sphagnum peat moss represents yet another type of hydrophobic substrate, different in structure from soil, and commonly used in commercial plant nurseries. Its commercial use in the absence of surfactants usually requires presoaking. However, information was desired on surfactant penetration of peat moss as received from supplier. The peat moss wetting data are less reproducible than those obtained with soil samples. Tarry balls usually formed on the surface of the ground peat moss, and penetration was uneven.

Hydrophilic-Lipophilic Balance

The hydrophilic-lipophilic balance (HLB) has been used to characterize nonionic surfactants as an aid in their application (11). Griffin's formula ($HLB = \text{weight percent oxyethylene content} \div 5$) has been used to calculate HLB for the compounds of Tables II and V, but the structure of methoxy ethers makes it difficult to define their hydrophilic limits.

HLB has a close relationship to solubility, which is often used to determine HLB experimentally. Many nonionic surface active agents seem to function best near their solubility limit. Therefore, it is not surprising that best wetting properties for nonionic compounds with the same hydrophobe are observed near the solubility limit (HLB 7-9). The measurement for the most effective wetting agents may be somewhat unreliable because actual solution concentration may be in doubt; some nonionics may not be soluble at 0.1% concentration at room temperature.

Wetting and Rewetting Characteristics

The data for the monoesters of polyethylene glycols and fatty acids are shown in Table II. In general it can be observed that, as the chain length of the acyl group is increased, more oxyethylene groups are required to obtain better wetting and rewetting efficiency. The diethylene glycol monoesters ($i = 2$) of octanoic and nonanoic acids ($n = 7$ and 8, respectively) possess the optimum balance between hydrophilic and lipophilic portions of the surfactant molecule, whereas for dodecanoic acid ($n = 11$) a pentaethylene glycol ester ($i = 5$) is required to achieve good wetting and rewetting. For the C_7 , C_8 , and C_9 compounds, the HLB values for monoesters of good wetting characteristics decrease with increasing chain length of the acyl group. If the chain length of the acyl group is extended beyond C_{11} , wetting properties become poor. Introduction of a double bond into the surfactant molecule also interferes with Glendora soil wetting but not rewetting.

There is a fair degree of correlation between Glendora soil data and the Draves test. The effect of structure variation on cotton wetting characteristics is less pronounced than wetting of Glendora soil. For example, the undecenoic esters wet cotton but not soil. However, on the whole, the trends are similar.

We surmised that the presence of diester might have an adverse effect on the wetting ability of monoesters. The test data from blends of mono- and diesters are given in Table III. The preparations shown in Table III are typical products of the above monoester synthesis (No. 1 and 2) or of the diester synthesis (No. 7 and blends of No. 1 or 2 with No. 7 to give mixtures No. 3-6. The data indicate that

wetting of Glendora soil and peat moss is greatly reduced with increasing diester content. Wetting of peat moss is more severely affected by diester content. The presence of as little as 11% of diester (No. 2) destroys both wetting and rewetting of peat moss, whereas Glendora soil can tolerate up to 27% of diester (No. 3), and the rewetting of Glendora soil is unaffected by the presence of even 61% of diester (No. 6). Since preparation No. 2 was the result of the synthesis of a "pure" monoester, it is obvious that fatty acid monoesters are of limited usefulness for peat moss applications.

In an effort to circumvent the problem of diester formation encountered with the polyethylene glycol monoesters, the monoesters of the monomethyl ethers of various polyglycols were prepared. The wetting data are shown in Table IV. The presence of a terminal methoxy instead of a hydroxyl group makes the compounds of this series less hydrophilic so that both wetting and rewetting characteristics are less satisfactory than those of the analogous polyethylene glycol monoesters. Although the presence of some diester impurity depresses the surface activity of monoesters, monoester samples containing small amounts of diester are still better wetting agents than esters of monomethyl ethers. The Draves test, Glendora soil, and peat moss data show some correlation, and the same general pattern visible in Table II is also apparent in Table IV. At a C_{13} or above chain length, wetting becomes inadequate, and as the carbon chain length is increased the number of oxyethylene groups also has to be increased to obtain wetting in a short period of time. However, in contrast to the monoesters of Table II, the esters of octanoic and nonanoic acids of Table IV possess poor wetting and only fair rewetting properties. None of the compounds of Table IV possesses the desired combination of rapid wetting and rewetting properties. It is apparent that the terminal hydroxyl group needs to be retained in the nonionic surfactant molecule in order to achieve good wetting.

Retention of a terminal hydroxyl group and freedom from contamination with byproducts containing two hydrophobic groups, such as diesters, is realized in the ether alcohols whose wetting and rewetting properties are shown in Table V. The ether alcohols at first glance might appear to be ideal wetting agents. However, as the table shows, the ether alcohols are no better wetting agents than the fatty acid monoesters of Table II. The main advantage of ether alcohols is their chemical stability. The length of the hydrophobic alkyl chain is critical for the ether alcohols. Only the decanol derivatives (No. 6-10) exhibit good wetting characteristics. The octanol derivatives have critical micelle concentration values in the range of 0.2-0.3%

(12), which is above the 0.1% concentration of the test solutions. The dodecanol derivatives No. 13, 14, and 14 wet cotton and are fair wetting agents on the other two substrates. A comparison between the decanol and dodecanol derivatives reveals again that with increasing alkyl chain length more oxyethylene groups are needed to attain wetting.

While the data of Table V show a somewhat erratic scatter, it is obvious that an ether alcohol with a C_{10} alkyl side chain and a relatively short hydrophilic group of two to three oxyethylene groups possesses the proper structure for good wetting on all three substrates (compounds No. 7 and 8). It is not surprising that the wetting requirements of peat moss are different from Glendora soil, since peat moss is mostly organic matter, while Glendora soil is sandy and has only a surface coating of very hydrophobic organic material.

Considering all the types of structures included in this study, best wetting is accomplished with fatty acids and alcohols having an alkyl chain of 9 or 10 carbon atoms with two or three oxyethylene groups attached and with the terminal hydroxyl group left intact. Difficulty in obtaining monoesters free from diester suggests that ether alcohols may be the preferred structure for wetting even though the best wetting was accomplished with certain pure monoesters.

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