Soil Wetting Agents from Fatty Nonionic Tertiary Amides: "Aromatic Adducts without Aminoester"

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Tertiary amides RCONR'K, where K is a hydroxy function and R,R' are aliphatic and/or aromatic groups, were oxyethylated to adducts containing 5, 10, 15, and 20 moles of ethylene oxide. The hydroxy function K was derived from $-CH_2CHR''OH$, $-(CH_2)_yOH$, or $(CH_2CH_2O)_zH$, where R″ is methyl, ethyl, or octyl, y is 4 or 6 methylenes, and z is 2 or 3 ethoxy groups. Structural variations in R,R' and K permitted the oxyethylation of aromatic amides with minimal aminoester formation. When K was diethoxy and R,R' were linear aliphatic groups, no aminoester accompanied oxyethylation. Minimal aminoester formed when R,R' were mixed aliphatic/aromatic or when R was phenethyl and R' aromatic. For aromatic amides where K was di- or triethoxy, large aminoester concentrations accompanied oxyethylation. For aromatic amides where K was $(CH_2)_4OH$ or $(CH_2)_6OH$ smaller aminoester concentrations were observed. For branched alphatic amides where R″ is methyl, ethyl, or octyl, aminoester concentration remained at the level found where R″ is hydrogen. The increasing size of R″ decreased solubility and Draves wetting times. The adducts of this study were wetting agents for Idylwild hydrophobic soil and cotton skeins. The availability of aromatic substrates should permit formulation of soil wetting agents with greater stability to biodegradation than would be expected with aliphatic substrates.

Water repellent soils occur on cultivated lands, forests, western chaparrals, citrus groves, and burned brushland (1,2). Organic residues generate the hydrophobic character on the soil surface (3). The property is associated with the plant growth-decay cycle and with bacterial and fungal growth. Water repellency leads to excessive runoff, soil erosion, and diminished crop yields. These effects can be controlled by either periodic cultivation or the use of soil amendments (4).

Nonionic surfactants were studied because they are physically adsorbed by soils, hence they should remain in the upper soil levels (5). They are unaffected by multivalent ions, and because they are surface active they should be effective at low concentration. Their rate of biodegradation when adsorbed on soils is proportional to the degree of adsorption (6) which is related to humus content. Nonionics promote water infiltration in water repellent soils but have little effect on hydrophilic soils (7).

Initial efforts by Weil *et al.* (8) showed that homogeneous oxyethylated linear acids and alcohols were wetting agents for cotton skeins, peat moss, and a hydrophobic soil with adducts containing ~ 10 carbons and 2-3 ethylene oxide (E.O.) groups. These adducts were characterized by good wetting and low water solubility. Subsequent studies of oxyethylated branched aromatic sulfonamides showed that polydisperse adducts are at least equally as good wetting agents as were homogeneous adducts (9). Aromatic sulfonamides add E.O. readily, giving adducts superior to a commercial soil wetting agent at low concentrations. A search for simpler substrates showed that aliphatic secondary amides (10) reacted incompletely with E.O. under low pressure conditions, while aromatic secondary amides formed adducts contaminated with an aminoester byproduct (11,12). The fact that aliphatic secondary amides added E.O. without forming any aminoester led us to examine the oxyethylation of aliphatic hydroxyethyl tertiary amides (13) $RCONR'CH_2CH_2OH$. When R and R' were linear groups, E.O. addition occurred, forming adducts whose ester/amide carbonyl ratio was ~ .25. Branched R groups reduced the aminoester considerably, giving adducts with ester/amide a carbonyl ratio of ~ .05. Polydisperse adducts from branched hydroxyethyl tertiary amides with short E.O. chain lengths showed instantaneous Draves wetting properties. This series was superior to a commercial wetting agent at a 0.025-0.1% level. These studies examined property-structure relationships resulting from variations in the nature of R groups. Work with adducts of branched aliphatic diamides RNHCOR'CONHR was conducted to examine the effects of manipulating the hydrophilic portion of these substrates (14). This effort showed: low pressure oxyethylation occurs only with a dihydroxyethyl substrate; excellent wetting properties, optimum surface activity, water solubility, and stability to aminoester formation occurred when R' was an ethylene group; all of these properties were diminished as the number of methylenes increased from 0 to 4.

These studies also showed that oxyethylated amides exhibited the most desirable spectrum of properties as soil wetting agents. However, adducts for actual use on hydrophobic soils should exhibit moderate stability to microbial activity. This can be realized only with a branched aromatic structure. The present program was aimed at developing aromatic systems which will add E.O. without aminoester formation and still have wetting properties comparable to aliphatic substrates. The approach involved altering the K portion of the tertiary amide, RCONR'K, to inhibit aminoester formation while incorporating aromatic structures in R and R'. K is a hydroxy bearing structure $-CH_2CHR''OH$, $-(CH_2)_yOH$ or $-(CH_2CH_2O)_zH$ for reaction with E.O. R″ is hydrogen, methyl, ethyl, or octyl while y is 0, 4, or 6 methylenes, and z is 2 or 3 ethoxy groups. The hydroxyamines were prepared as follows:

$$R'NH_2 + R''CHCH_2 \rightarrow R'NHCH_2CHOHR'' \quad (1)$$

$$R'NH_2 + Cl(CH_2)_y\,OH \rightarrow R'NH(CH_2)_y\,OH \quad (2)$$

$$R'NH_2 + Cl(CH_2CH_2O)_z\,H \rightarrow R'NH(CH_2CH_2O)_z\,H \quad (3)$$

The reaction of these amino alcohols with an acid chloride gave the required tertiary amide (15). The addition of low pressure E.O. at 80C/50 psi using≤0.1 mol/% solid KOH formed adducts with 5-20 moles E.O. per mole of amide.

EXPERIMENTAL

Materials and Apparatus. Epoxyalkanes, chloro alcohols, chloroethoxy alcohols, acid chlorides, and various amines were obtained from Aldrich Chemical Co., Metuchen, New Jersey or the Eastman Kodak Co., Rochester, New York. Various solvents and reagents were used as received. All synthesized intermediates were purified by either a simple distillation or fractionation through a 2′ spinning band column @ ~ 0.2 mm. Oxyethylations were performed in half liter, round bottomed pressure flasks equipped with a thermometer well and an aerosol pressure coupling #110-585 from Lab-Crest Scientific Division, Fischer Porter Co., Warminister, Pennsylvania. The aerosol coupling was connected to a 0-60 psi pressure gauge and a pressure release valve. Similarly equipped glass pressure tubes with a capacity of ~ 90 ml were used in oxyethylations of small volumes of reactants. A teflon-coated bar magnet served as a stirrer. Infrared spectra were obtained with a Perkin Elmer 1310 microprocessor controlled infrared spectrophotometer. Nonaqueous potentiometric titrations were performed with a Beckman Zeromatic pH meter using glass and calomel electrodes in an alcohol-glycol solvent. Surface tension measurements were made with a duNoüy tensiometer.

Idylwild hydrophobic soil was obtained through the courtesy of J. Letey and co-workers from the University of California at Riverside. This soil is burned over brushland sandy loam from the San Jacinto mountains near Idylwild, California. The soil was sieved to pass a #10 screen, air dried, and bottled to maintain a constant moisture level.

Substituted Alkylethanolamines. These intermediates were prepared using the same general procedure as described for the reaction between 2-ethylhexylamine and ethylene oxide (13). Elemental analyses agreed with theory by ± 0.3%. Neutral equivalents indicated purities of ~ 97%.

2 (2-N-n-butylphenylaminoethoxy) ethanol. 37.5g, 0.3 mole 2-(2-chloroethoxy)-ethanol was added dropwise to a 0.5 l R.B. flask containing 179g, (1.2 moles) p-n-butylaniline with vigorous stirring at 120C over a 2 hour period. The reaction mixture was stirred for an additional 4-6 hours at this temperature and was then diluted with 175 ml of 1,2-dichloroethane. The black reaction mixture was treated with 100 ml distilled water containing 0.35 mole sodium hydroxide. After separating the layers, the organic layer was washed 4x with 75 ml portions of distilled water and filtered by gravity. Excess p-butylaniline was distilled at 0.4 mm, leaving 75 g of black residue. Fractionation of the residue through the 24″ spinning band column gave 56 g of off-white distillate which darkened upon standing. The neutral equivalent corresponded to a 97% purity. The amines in this class generally showed purities better than 95%.

N-(ω-hydroxyalkyl)-p-butylaniline. The procedure just described for the preparation of di- and triethoxyamines was used to synthesize these intermediates. 4-chlorobutanol and 6-chlorohexanol yielded intermediates whose neutral equivalents indicated a purity of ~ 90%.

Monomer Tertiary Amide. These substrates were prepared by adding the required acid chloride to one of the following amine types described in the preceding sections: RNHCH$_2$CHR′OH, RNH(CH$_2$CH$_2$O)$_x$ H, or RNH(CH$_2$)$_x$ OH. The procedure was previously described (14) and was used with some modification. The acid chloride was added in 30 minutes at ~ 15-20C. The slurry was stirred at room temperature for 2 hours and filtered. After removal of solvent from the washed organic phase, all crude products were distilled at a reduced pressure of ~ 0.1 mm. Elemental analyses were within ± 0.3% of theory.

N-(2-hydroxyalkyl)-2-Ethylhexylamine

RNHCH$_2$CHR′OH

R	R′	Reaction Time, Hrs., Temp C		Product b.p. C/Press, mm.	Yield %
2-ethylhexyl	octyl	20	150	115-19/0.1	70
2-ethylhexyl	ethyl	24	135	72-5/0.3	72
2-ethylhexyl	methyl	5	70-120	67-70/0.3	70

N-Di- and Triethoxyamines

RNH(CH$_2$CH$_2$O)$_x$ H

R	x	b.p. C	Press., mm.	Yield %
octyl	2	97-100	0.1	71
p-butylphenyl	2	128-33	0.1	79
p-butylphenyl	3	153-56	0.1	74

ω-hydroxyalkylanilines

$RNH(CH_2)_x OH$

R	x	b.p. C	Press., mm.	Yield %
p-butylphenyl	4	135-40	0.3	32
p-butylphenyl	6	150	0.3	65

Monomer Tertiary Amide

RCONR'K

R	R'	K	b.p. C	Press, mm.	Yield %
3-heptyl	2-ethylhexyl	$-CH_2CHOHC_8H_{17}$	used as isolated		
3-heptyl	2-ethylhexyl	$-CH_2CHOHC_2H_5$	128-33	0.15	83
3-heptyl	2-ethylhexyl	$-CH_2CHOHCH_3$	125- 8	0.20	76
heptyl	octyl	$-(CH_2CH_2O)_2H$	160- 4	0.10	81
p-t-butylphenyl	octyl	$-(CH_2CH_2O)_2H$	205-10	0.15	84
heptyl	p-butylphenyl	$-(CH_2CH_2O)_2H$	180- 5	0.10	80
3-heptyl	p-butylphenyl	$-(CH_2CH_2O)_2H$	173- 8	0.20	75
p-t-butylphenyl	p-butylphenyl	$-(CH_2CH_2O)_2H$	205-15	0.15	74
2-phenethyl	p-butylphenyl	$-(CH_2CH_2O)_2H$	207-13	0.10	75
p-t-butylphenyl	p-butylphenyl	$-(CH_2CH_2O)_3H$	217-30	0.10	72
p-t-butylphenyl	p-butylphenyl	$-(CH_2)_6OH$	230- 5	0.15	77
p-t-butylphenyl	p-butylphenyl	$-(CH_2)_4OH$	208-12	0.15	75

Oxyethylation of tertiary amide. This procedure was used as previously described (13).

Evaluation. A modified version of the Draves test (16) was used to determine the wetting profile of a given series of E.O. adducts. The procedure measures the time in seconds for a standard 5 g cotton skein to sink in a 0.1% solution of surfactant at room temperature. The water drop penetration test (17) was developed as a screening procedure to evaluate the behavior of wetting agents on hydrophobic soils. A cylindrical polyethylene cup (dia. 1.5 cm × 1.0 cm) was half filled with Idylwild soil. The sample was leveled and given a slight concave surface. Surfactant solution (0.05 ml) or distilled water was applied to the surface and the time required for disappearance of free liquid was measured. Wetting agents were evaluated at 0.1, 0.01, and 0.001% (w/v) solution. Rewet time was measured after applying 0.05 ml of distilled water to the same soil surface. Two approaches were used: wetted soil samples were dried overnight at room temperature and then rewetted, or they were dried for 30 minutes on a steam bath, equilibrated for 1 hour and then rewetted. The results were generally comparable.

Cloud point was determined on 1% aqueous solutions of E.O. adduct. A turbid sample solution was cooled until it became clear. The clear sample was warmed to incipient turbidity. The solution temperature was recorded as the cloud point. The procedure was then repeated to duplicate the value. Surface tension values were determined on 0.1% aqueous solutions and the values were corrected to 25C.

RESULTS AND DISCUSSION

Synthesis. N-(2-hydroxyalkyl)-2-ethylhexylamines were obtained as colorless oils in ~ 70% yields by reacting 1,2-epoxyalkanes with 2-ethylhexylamine under low pressure conditions (<50 psi). In contrast to the high reactivity of ethylene oxide, 1,2-epoxyalkanes with alkyl groups larger than methyl required ~ 24 hours at 135-150C for complete reaction. Di- and triethoxyamines were isolated in 70-80% yields by the dropwise addition of the required chloroethylene glycol to an aliphatic or aromatic amine in a 1:4 molar ratio, respectively. The additions were performed neat at 100-120C. An essentially analogous procedure was used to prepare ω-hydroxyalkylanilines in 30-65% yields. All the amine intermediates were purified by distillation through the spinning band column.

Monomer tertiary amides were viscous oils obtained in ~ 75% yields. Because of their high molecular weight these substrates were simply distilled at reduced pressure under a nitrogen atmosphere. This simple procedure was sufficient to remove most, and usually all, of the residual aminoester found in these preparations (Table 1). As mentioned in earlier studies (13), this purification was necessary to remove inhibitors to oxyethylation. Generally a purified substrate always added E.O. more readily than the crude product. Surprisingly, the tertiary amide derived from 1,2-epoxydecane could not be distilled without forming large amounts of the ester contaminant. In this instance, it appears that the threshold of thermal

TABLE 1

Carbonyl Ratios of Tertiary Amide Adducts $RCONR'(K)(E.O.)_xH$

					Carbonyl Ratio* at x values		
	R	R′	K	Initial x**	Initial	10	20
1)	C_7H_{15}	C_8H_{17}	$(E.O.)_2$	2	0.0	0.0	0.0
2)	p-t-$C_4H_9\phi$	C_8H_{17}	$(E.O.)_2$	2	.06	.05	.04
3)	C_7H_{15}	p-n-$C_4H_9\phi$	$(E.O.)_2$	2	0.0	.09	.08
4)	p-t-$C_4H_9\phi$	p-n-$C_4H_9\phi$	$(E.O.)_2$	2	.10	.66	2.1
5)	p-t-$C_4H_9\phi$	p-n-$C_4H_9\phi$	$(E.O.)_3$	3	.01	.58	3.6
6)	ϕCH_2CH_2	p-n-$C_4H_9\phi$	$(E.O.)_2$	2	0.0	.03	.04
7)†	p-t-$C_4H_9\phi$	p-n $C_4H_9\phi$	H	0	0.0	1.4	1.4
8)	p-t-$C_4H_9\phi$	p-n-$C_4H_9\phi$	$(CH_2)_4O$	1	0.0	.27	.37
9)	p-t $C_4H_9\phi$	p-n-$C_4H_9\phi$	$(CH_2)_6O$	1	0.0	.06	.25
10)†	3-C_7H_{15}	2-$C_2H_5C_6H_{12}$	CH_2CH_2O	1	.02	.05	.04
11)	3-C_7H_{15}	2-$C_2H_5C_6H_{12}$	CH_2CHCH_3O	1	.02	.05	.03
12)	3-C_7H_{15}	2-$C_2H_5C_6H_{12}$	$CH_2CHC_2H_5O$	1	.03	.07	.06
13)	3-C_7H_{15}	2-$C_2H_5C_6H_{12}$	$CH_2CHC_8H_{17}O$	1	0.0	.05	.04

*Ester/amide carbonyl ratio measured @ ~ 1730/1630 cm^{-1}

**Substrate E.O. chain length.

†Series 7 and 10 from references 10 and 13, respectively.

stability for the amide configuration was reached. General infrared spectral assignments

$$RCONR'CH_2CHC_8H_{17}OH \overset{\Delta}{\rightleftharpoons} R'NHCH_2CHC_8H_{17}OCOR$$

for the amides and adducts were previously described (10,13). It was noted that as the degree of oxyethylation increased, the OH band drifted to shorter wavelengths, as did the amide carbonyl band, but to a much lesser degree.

Oxyethylated Amides. Table 1 summarizes the ester/amide carbonyl ratios for adducts where K is hydrogen, substituted ethoxy, di- or triethoxy, or a hydroxy-alkyl group. Initial x corresponds to the substrate E.O. chain length. Carbonyl ratios are given for initial x, when x equals 10 and 20, and are a relative measure of aminoester content. When R and R′ are linear aliphatic groups and K is diethoxy, E.O. addition occurred without forming any ester carbonyl. If K had been ethoxy (13), a fair amount of ester carbonyl would have formed. In adduct series 2 and 3 some ester carbonyl forms when either R or R′ is an aromatic group. When both groups are aromatic, as in series 4 and 5, a progressive increase in ester carbonyl accompanies oxyethylation. Why this occurs when the initial x is greater than one is not known. The formation of progressively larger hemiacetal ring intermediates (11) seems unlikely. However, if K had been a simple ethoxy group, ester carbonyl would have formed even more readily. Adduct series 6 shows that ester carbonyl can be largely inhibited in an aromatic structure if R is a phenethyl group. Adduct series 7-9 show the effect upon carbonyl ratio when K is hydrogen, butyloxy, or hexyloxy. E.O. addition to a simple secondary aromatic amide (adduct 7) results in immediate formation of ester carbonyl. Increasing the methylene chain of K to butyloxy or hexyloxy does give a progressive decrease in the carbonyl ratio during oxyethylation. When R and R′ are branched 8 carbon aliphatic groups, as in series 10-13, and the ethoxy group of K contains even larger alkyl groups, we observe essentially no change in carbonyl ratio during oxyethylation. For series 11, plots of surface tension versus E.O. chain length showed minimal surface tension values at short chain lengths paralleling the results previously described for series 10. However, plots for series 12 and 13 over chain lengths 5-20 and 10-20, respectively, showed no change in surface tension.

Figure 1 shows Draves wetting profiles for branched aliphatic adducts where R″ is hydrogen, methyl, ethyl, or octyl. Wetting time in seconds is plotted against E.O. chain length x + 1. Contact angle data were not applicable here because the approach does not differentiate the wetting ability of members in a given series. Instantaneous wetting times are retained for R″ being hydrogen or methyl, which is an overall improvement for the series with an ethyl group and a complete loss of wetting ability when R″ is octyl. Figure 2 shows wetting profiles of aromatic adducts derived from the secondary amide where R is butyloxy or hexyloxy. E.O. addition to the secondary amide results in immediate aminoester formation and very poor wetting properties. When R is butyloxy, reduced ester carbonyl is found and an improved wetting profile is obtained. With a hexyloxy group a further decrease in ester carbonyl occurs, but wetting properties are deteriorating due to the longer methylene chain.

Figure 3 summarizes similar wetting profiles for adducts derived from di- and triethoxy substrates. The first three profiles show that insertion of an aromatic group does not change the basic wetting pattern from an aliphatic series. When both R groups are aromatic and n is 2 or 3, the effects of high aminoester content and low solubility results in poor wetting properties for both series. Insertion of a phenethyl group in R yields a profile with fairly attractive wetting behavior at a short E.O. chain length along with minimal ester carbonyl.

The relation between cloud point and E.O. chain length for hindered aliphatic adducts where R″ is hydrogen, methyl, ethyl, or octyl is given in Figure 4. In these series where E.O. was added in 5 mole increments, all adducts with a chain length of 5 had cloud points below 1C. These series are shown in order of decreasing solubility and

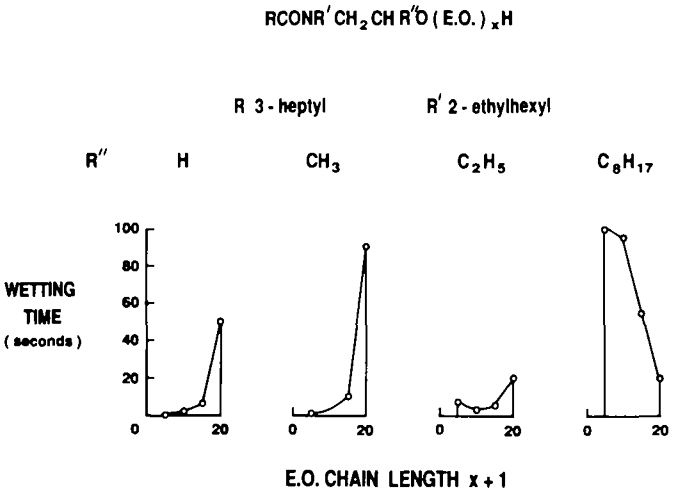

FIG. 1. Draves wetting profiles of aliphatic tertiary amide adducts.

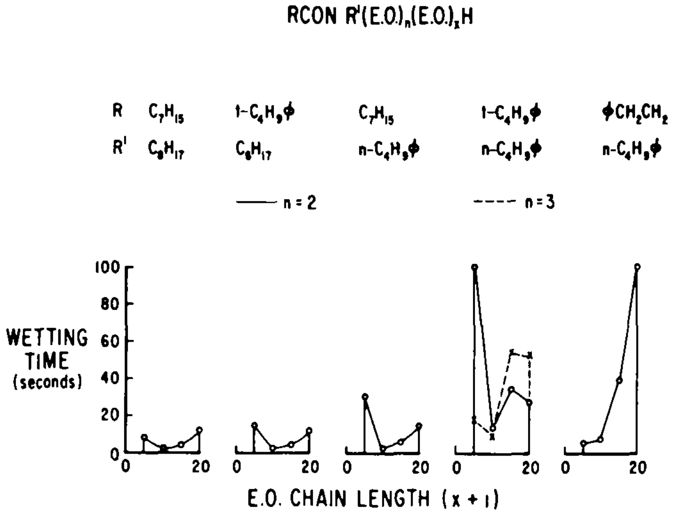

FIG. 3. Draves wetting profiles of di- and triethoxy derived adducts.

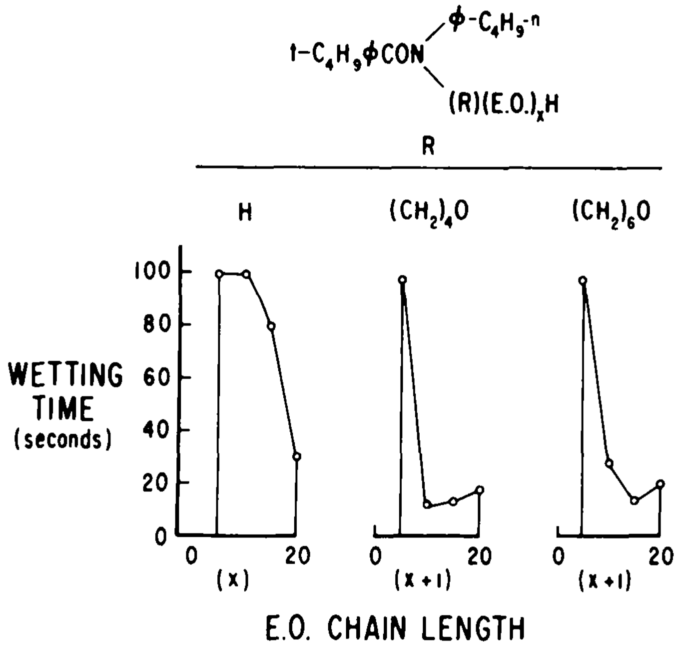

FIG. 2. Draves wetting profiles of hydroxyalkyl derived aromatic adducts.

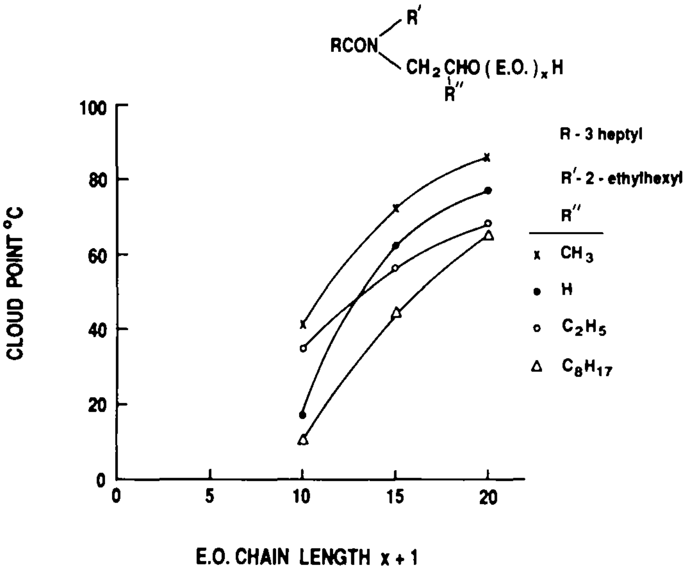

FIG. 4. Aqueous solubility of aliphatic tertiary amide adducts.

show normal solubility which increases with E.O. chain length. The greater solubility of the series where R'' is methyl is probably a reflection of its lower packing density relative to the more symmetrical unsubstituted series. Similar solubility curves are observed in Figure 5 for purely aromatic adducts where R is either hydrogen or an alkyloxy group with 4 or 6 methylenes. The adduct series are listed in order of decreasing solubility and increasing carbonyl ratio at E.O. chain length 20. E.O. addition to the substrates where R is hexyloxy or butyloxy exhibit normal solubility curves in spite of carbonyl ratios of ~ 0.30. Oxyethylation of the free amide leads to adducts with high carbonyl ratios and correspondingly poor solubility. Figure 6 shows cloud point curves for adducts from di- and triethoxy substrates listed in order of decreasing solubility with the corresponding carbonyl ratio for E.O. chain length of 20. Normally chain extension leads to increasing solubility as reflected by increasing cloud points. The results show that normally expected solubility curves are associated with low carbonyl ratios. The last two series with high carbonyl ratios reflect the formation of hydrophobic adducts by E.O. addition to the amino group. These series should have identical solubility curves. However, the differences reflect the complex nature of the aminoester contaminants and the oxyethylation of these systems.

Table 2 shows the drop penetration wetting times for Idylwild hydrophobic soil by aromatic adducts where the E.O. chain length ranges from 5 to 20. The samples derived from diethoxy substrates differ considerably in ester carbonyl, Draves wetting, and water solubility. However, the differences are not particularly apparent from these values. The third series, where R is a heptyl group, showed the best overall Draves wetting profile and appears to confirm good wetting even at the 0.01% level.

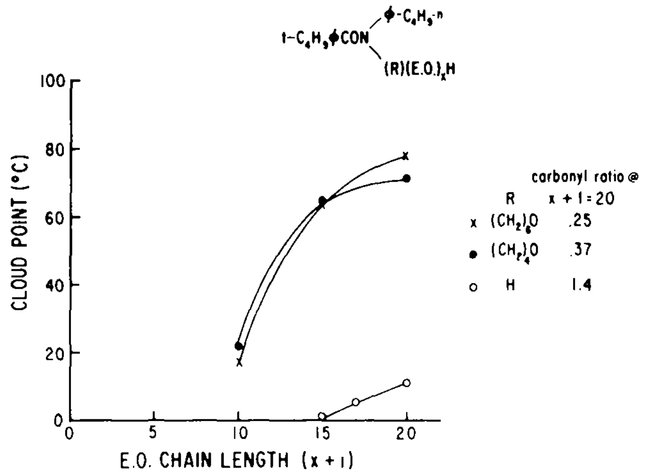

FIG. 5. Aqueous solubility of amide adducts from aromatic substrates.

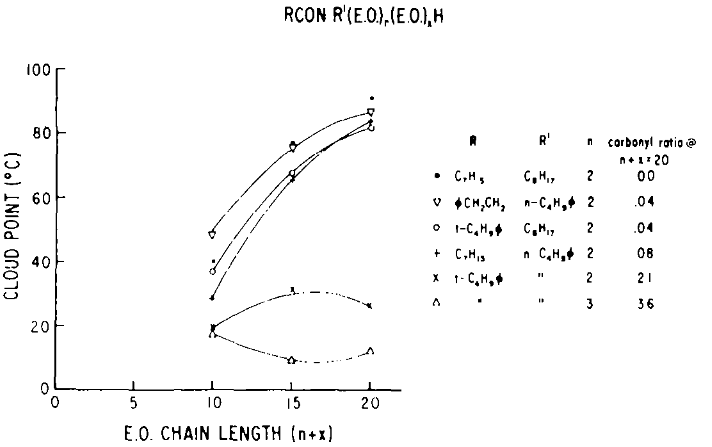

FIG. 6. Aqueous solubility of di- and triethoxy derived amide adducts.

The first series shows comparable results. Relative to the distilled water controls the results suggest positive effects even at 0.001%. Rewet times are generally shorter than wet times, but the same behavior occurs with the controls. At a given concentration there is no obvious difference between series or adducts in a given series.

Similar results are shown in Table 3 for branched aliphatic adducts derived from substituted hydroxyethyl tertiary amides where R″ increases from hydrogen to an octyl group. These series of adducts have very low carbonyl ratios, with both wetting properties and solubility decreasing as the size of R″ increases. The deterioration of drop penetration wetting time appears to be following the same course for these results at the 0.1% level. The same general comments made for the results in Table 2 apply to these data.

In conculsion, oxyethylation of diethoxy substrates RCONR′(E.O.)$_2$H, where R and R′ are linear aliphatic groups, gives adducts free of aminoester contaminants. This is a new approach to uncontaminated aliphatic amide adducts (13). Oxyethylation of the same diethoxy substrates where either R or R′ are aromatic, or R is phenethyl and R′ aromatic, give adducts with low ester carbonyl ratios and good to moderate wetting properties. For di- or triethoxy substrates where R and R′ are aromatic groups, oxyethylation proceeds with increased aminoester contamination. This is reflected by poor water solubility and wetting properties. This behavior is mitigated noticeably in the oxyethylation of aromatic substrates RCONR′K where K is H, $(CH_2)_4OH$ and $(CH_2)_6OH$. As the number of methylenes increased, aminoester contamination and water solubility decreased, while wetting properties improved. Adduct series derived from substituted hydroxyethyl amides RCONR′CH$_2$CHR″OH, where R″ was H, CH_3, C_2H_5, or C_8H_{17}, show no change in ester carbonyl but decreasing wetting properties and water solubility as the size of R″ increases. The drop pene-

TABLE 2

Drop Penetration Wetting of Idylwild Soil by Aromatic Adducts

RCONR′(E.O.)$_2$(E.O.)$_x$H

R R′	φCH$_2$CH$_2$ p n-C$_4$H$_9$φ		p-t-C$_4$H$_9$φ p n-C$_4$H$_9$φ		C$_7$H$_{15}$ p-n-C$_4$H$_9$φ		H$_2$O Control	
			Time Seconds @ 0.1%					
x + 2	Wet	Rewet	Wet	Rewet	Wet	Rewet	Wet	Rewet
5	8	4	9	75	4	1	880	60
10	9	1	10	2	2	3	880	60
15	4	1	23	8	5	3	880	60
20	5	1	22	3	6	2	880	60
				@0.01%				
5	140	33	135	45	120	29	500	80
10	165	10	120	70	35	15	500	80
15	160	10	130	15	40	22	500	80
20	195	10	160	28	90	29	500	80
				@0.001%				
5	225	33	190	33	205	85	925	60
10	265	24	250	180	240	43	925	60
15	240	140	300	45	280	40	925	60
20	305	38	480	28	250	48	925	60

TABLE 3

Drop Penetration Wetting of Idylwild Soil by Branched Adducts

$RCONR'CH_2CHR''O(E.O.)_xH$*

R''	H		CH_3		C_2H_5		C_8H_{17}		Water Control	
					Time Seconds @ 0.1%					
x + 1	Wet	Rewet	Wet	Rewet	Wet	Rewet	Wet	Rewet	Wet	Rewet
5	2	2	1	2	6	2	130	12	970	110
10	3	1	1	1	3	2	7	10	970	110
15	2	1	2	1	2	1	31	6	970	110
20	3	1	3	2	3	1	23	4	970	110
					@0.01%					
5	200	12	40	18	115	30	360	665	1075	190
10	150	15	65	9	85	40	315	90	1075	190
15	250	17	70	7	120	45	320	40	1075	190
20	210	19	110	10	175	25	270	55	1075	190
					@0.001%					
5	270	40	170	34	200	92	480	310	710	190
10	410	300	180	15	320	55	735	180	710	190
15	410	180	220	37	310	160	960	85	710	190
20	190	420	140	57	420	70	360	90	710	190

*R and R′ are 3-heptyl and 2-ethylhexyl, respectively.

tration wetting procedure is useful in determining gross differences in a homologous series but is of little value in distinguishing between separate adduct series. Higher ester/amide carbonyl ratios are associated with polydisperse adducts showing low solubility and poor wetting properties.

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