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Effect of the Temperature on the Conductivity of Sodium Bis(2-ethylhexyl)sulfosuccinate + 2,2,4-Trimethylpentane + Water Microemulsions in the Presence of Ureas and Thioureas

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The effect of the temperature on the conductivity of ternary systems of sodium bis(2-ethylhexyl)-sulfosuccinate + 2,2,4-trimethylpentane + water has been studied. The effect of the presence of organic additives—urea, methylurea, 1,3-dimethylurea, 1,1-dimethylurea, tetramethylurea, thiourea, methylthiourea, 1,3-dimethylthiourea, and tetramethylthiourea—on the variation of the conductivity with the temperature in these ternary systems has also been studied.

Introduction

Microemulsions are transparent isotropic dispersions of an immiscible organic compound in water in the presence of a surfactant (Pileni, 1989). They have been described as consisting of spherical droplets of a disperse phase separated from a continuous phase by a film of surfactant (Schulman et al., 1959). In this work will be studied the microemulsions formed by sodium bis(2-ethylhexyl)sulfo-succinate + 2,2,4-trimethylpentane + water (AOT + isooctane + water). These systems present a great interest from the point of view of pure chemistry as well as from that of applied chemistry due to the fact that the systems have a great potential as solubilizators (Mittal, 1977; Elworthy et al., 1969; McBain and Hutchison, 1955; García-Rio et al., 1995, 1996), permitting an important number of industrial applications (Rieger, 1977; Datyner, 1983; Kuhn, 1963).

When the temperature increases, the conductivity of these systems increases gradually until a determined temperature is reached from which a sudden increase of the conductivity is produced. This phenomenon is known as electrical percolation, and the temperature at which it is produced is known as the percolation threshold or temperature of percolation.

An interesting group of papers (Jada et al., 1989, 1990; Lang et al., 1990) have demonstrated the existence of a correlation between the electrical percolation and the rate constant of mass transfer process between droplets. This process of mass transfer is essential in order to determine the chemical reactivity in water/oil microemulsions.

The possibility that small quantities of additives can modify the value of the percolation threshold has been documented in the literature (Matthew et al., 1988). These studies have established that additives that make surfactant membranes more rigid—such cholesterol—hinder the appearance of electrical percolation, while those that make the membrane more flexible—such gramicidine—favor it. These results together with others (Jada et al., 1989, 1990; Lang, 1990) indicate that the process of electrical percolation it not associated with the formation of bicontinuous structures in the microemulsions, but the structure of discreet droplets is maintained. Probably, the number of collisions increases suddenly in the vicinity of the percolation point. This behavior allows the formation of clusters of droplets between which channels that permit the flow of mass between droplets are opened.

The aim of this work is to measure the electrical conductivity of ternary systems with different additives, at different temperatures. From these data we are able to get the temperature at which the electrical percolation is produced.

Experimental Section

The aqueous solutions of urea (U), methylurea (MU), 1,3-dimethylurea (1,3-DMU), 1,1-dimethylurea (1,1-DMU), tetramethylurea (TTMU), thiourea (TU), methylthiourea (MTU), 1,3-dimethylthiourea (1,3-DMTU), and tetramethylthiourea (TTMTU) were prepared with distilled–deionized water. The solutes were supplied by Merck and Sigma, and all of then were of the maximum purity commercial available (>99%). They all were employed without further purifications.
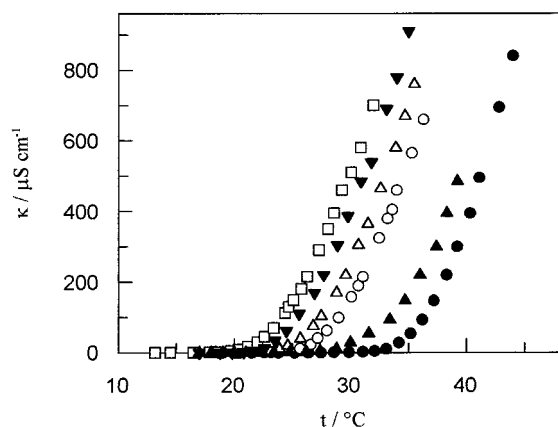
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Table 1. Electrical Conductivity Values at Different Temperatures, without Additive and in the Presence of Different Concentrations of Ureas and Thioureas in Sodium Bis(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-Trimethylpentane + Water Microemulsions ([AOT] = 0.5 mol·dm^{-3}, [H$_2$O]/[AOT] = 22)

$t/°C$	$\kappa/\mu S\cdot cm^{-1}$	$t/°C$	$\kappa/\mu S\cdot cm^{-1}$	$t/°C$	$\kappa/\mu S\cdot cm^{-1}$	$t/°C$	$\kappa/\mu S\cdot cm^{-1}$	$t/°C$	$\kappa/\mu S\cdot cm^{-1}$	$t/°C$	$\kappa/\mu S\cdot cm^{-1}$
				Without Additive							
23.80	0.37	28.60	0.82	32.10	5.00	35.20	54.00	38.30	220.00	41.10	495.00
25.20	0.43	29.90	1.29	33.10	10.90	36.20	93.00	39.20	300.00	42.80	695.00
26.40	0.51	31.10	2.60	34.20	28.00	37.20	147.00	40.30	395.00	44.00	840.00
27.50	0.62										
				[thiourea] = 0.04 mol·dm^{-3}							
21.00	0.26	25.40	0.44	30.70	4.55	34.10	44.00	38.40	240.00	42.20	590.00
21.50	0.27	27.40	0.79	31.40	7.50	35.20	69.00	39.90	380.00	43.50	750.00
23.00	0.31	28.20	1.08	32.20	13.20	36.20	112.00	41.00	470.00	44.60	910.00
24.00	0.35	29.90	2.45	33.00	28.50	37.10	160.00				
				[thiourea] = 0.2 mol·dm^{-3}							
16.70	0.22	20.20	0.42	23.20	1.60	27.20	41.00	30.70	190.00	33.60	405.00
18.70	0.30	20.50	0.46	24.80	5.70	28.00	62.50	31.10	215.00	34.00	460.00
19.00	0.33	21.50	0.68	25.70	11.70	29.00	99.00	32.50	325.00	35.30	565.00
19.90	0.38	22.60	1.14	26.60	23.00	30.10	158.00	33.20	380.00	36.30	660.00
				[methylthiourea] = 0.2 mol·dm^{-3}							
18.40	0.42	21.60	1.60	24.60	20.00	27.50	104.00	30.70	305.00	33.90	580.00
19.40	0.55	22.40	3.05	25.70	40.05	28.80	170.00	31.50	365.00	34.70	670.00
20.50	0.90	23.90	11.20	26.80	75.50	29.60	220.00	32.60	465.00	35.50	760.00
				[1,3-dimethylthiourea] = 0.04 mol·dm^{-3}							
21.70	0.30	25.70	0.62	30.60	9.20	34.70	114.00	37.50	255.00	40.20	490.00
22.30	0.32	26.80	0.97	31.20	15.25	35.60	145.00	38.40	320.00	41.30	560.00
23.30	0.36	27.80	1.46	32.80	47.50	36.80	210.00	39.50	405.00	42.40	690.00
24.10	0.42	29.40	4.10	33.50	62.00						
				[1,3-dimethylthiourea] = 0.2 mol·dm^{-3}							
13.20	0.24	18.60	1.68	22.00	30.05	24.70	130.00	27.30	290.00	30.10	510.00
14.50	0.30	19.70	4.40	22.60	45.00	25.10	148.50	28.10	350.00	30.90	580.00
16.50	0.54	20.40	8.00	23.40	69.00	25.80	180.25	28.60	395.00	32.00	700.00
17.90	1.05	21.20	16.75	24.40	112.00	26.30	215.00	29.30	460.00		
				[tetramethylthiourea] = 0.04 mol·dm^{-3}							
20.30	0.25	23.10	0.34	25.80	0.53	29.60	2.25	33.50	21.50	37.00	138.00
21.00	0.27	23.80	0.38	26.30	0.61	30.20	3.10	35.10	47.50	38.10	245.00
22.10	0.30	24.90	0.44	28.00	1.02	31.30	6.90	35.70	70.00	39.00	320.00
				[tetramethylthiourea] = 0.2 mol·dm^{-3}							
10.80	0.19	16.40	0.37	21.10	2.05	24.40	20.00	29.40	202.50	31.60	360.00
12.30	0.22	17.70	0.52	22.20	3.90	25.50	43.00	29.80	227.50	32.20	385.00
13.40	0.24	18.40	0.61	22.40	4.65	26.40	65.00	30.30	265.00	33.20	445.00
14.30	0.27	19.50	0.88	23.10	7.20	27.20	96.00	30.80	295.00	34.00	500.00
15.40	0.32	20.10	1.16	23.20	8.30	28.10	132.00	31.20	327.50		
				[urea] = 0.156 mol·dm^{-3}							
8.90	0.20	14.70	0.60	19.20	6.00	22.00	66.00	25.70	200.00	28.80	417.00
10.30	0.24	17.30	1.40	19.50	13.20	23.70	110.00	26.50	250.00	29.60	493.00
12.20	0.28	17.80	2.63	20.40	30.50	24.70	145.00	27.80	340.00	30.80	590.00
13.00	0.36	18.60	4.95	21.50	43.00						
				[urea] = 0.234 mol·dm^{-3}							
7.50	0.28	10.40	1.67	13.00	19.50	18.00	185.00	23.90	590.00	28.70	1060.00
7.70	0.30	10.80	2.65	13.50	27.50	19.10	245.00	25.00	680.00	29.50	1140.00
8.20	0.34	11.30	4.10	14.00	38.50	19.90	290.00	26.30	800.00	30.50	1260.00
8.50	0.40	11.60	6.20	14.80	60.00	20.90	360.00	27.20	900.00	31.70	1390.00
9.60	0.82	12.00	8.60	15.90	94.00	21.80	423.00	28.10	980.00	33.10	1560.00
10.00	1.10	12.40	12.20	17.00	134.00	23.00	510.00				
				[tetramethylthiourea] = 0.2 mol·dm^{-3}							
15.80	0.41	19.80	2.01	23.10	49.00	27.10	267.00	29.80	480.00	33.20	900.00
16.40	0.47	20.60	4.80	24.00	82.00	27.90	333.00	30.80	570.00	34.40	1050.00
18.20	0.80	21.30	11.10	25.00	130.00	29.10	435.00	31.80	730.00	35.20	1170.00
19.20	1.26	22.20	25.30	26.00	170.00						
				[methylurea] = 0.2 mol·dm^{-3}							
17.00	0.26	21.00	0.51	24.80	1.29	28.80	10.90	33.20	93.00	37.40	300.00
18.00	0.30	22.20	0.62	26.20	2.60	30.00	28.00	34.70	147.00	38.30	395.00
19.00	0.36	23.40	0.82	27.40	5.00	31.60	54.00	36.00	220.00	39.20	485.00
20.00	0.42										
				[methylurea] = 0.4 mol·dm^{-3}							
9.20	0.18	19.00	4.65	23.20	99.00	26.90	330.00	30.90	720.00	35.40	1270.00
13.50	0.32	21.00	27.20	24.40	160.00	28.10	430.00	32.50	920.00	36.80	1470.00
16.20	0.61	21.60	41.00	25.80	250.00	29.70	600.00	34.00	1100.00	38.70	1750.00
17.20	1.02										
				[methylurea] = 0.81 mol·dm^{-3}							
3.00	0.26	6.00	2.23	9.50	54.00	14.60	305.00	21.20	880.00	28.00	1700.00
3.40	0.31	6.40	3.67	10.20	74.00	15.60	373.00	22.90	1080.00	29.80	1920.00
4.50	0.48	7.00	7.00	11.90	148.00	18.00	570.00	23.90	1180.00	31.50	2150.00
4.80	0.65	7.60	14.10	12.80	193.00	19.40	695.00	26.30	1480.00	33.20	2400.00
5.60	1.43	8.10	21.00								
				[1,3-dimethylurea] = 0.2 mol·dm^{-3}							
3.60	0.12	11.00	0.20	18.80	0.82	23.50	35.50	28.90	305.00	34.00	780.00
5.00	0.13	12.10	0.22	19.80	1.24	24.50	63.00	29.80	387.00	35.00	910.00
6.30	0.14	14.30	0.28	20.80	2.44	25.60	111.00	30.90	485.00	36.00	1030.00
8.20	0.16	15.50	0.33	21.70	6.00	26.90	170.00	31.80	540.00	37.00	1150.00
9.60	0.18	17.00	0.42	22.60	13.00	27.70	221.00	33.10	690.00		

Table 1 (continued)

$t/°C$	$\kappa/\mu S \cdot cm^{-1}$	$t/°C$	$\kappa/\mu S \cdot cm^{-1}$	$t/°C$	$\kappa/\mu S \cdot cm^{-1}$	$t/°C$	$\kappa/\mu S \cdot cm^{-1}$	$t/°C$	$\kappa/\mu S \cdot cm^{-1}$	$t/°C$	$\kappa/\mu S \cdot cm^{-1}$
					[1,3-dimethylurea] $= 0.4$ mol·dm^{-3}						
3.90	0.14	9.10	0.26	14.10	2.07	19.10	134.00	26.00	740.00	32.60	1610.00
5.90	0.17	10.70	0.36	15.20	7.20	21.70	310.00	28.00	1000.00	35.20	2000.00
7.10	0.20	11.60	0.45	16.10	23.00	22.80	405.00	29.60	1200.00	37.00	2290.00
8.30	0.23	13.20	1.01	18.00	81.00	24.20	540.00	31.20	1410.00		
					[1,1-dimethylurea] $= 0.2$ mol·dm^{-3}						
3.90	0.12	10.40	0.17	18.80	0.34	24.80	2.15	29.90	98.00	37.60	760.00
5.60	0.13	12.10	0.19	20.00	0.41	25.90	5.20	31.60	190.00	39.20	970.00
6.90	0.14	13.50	0.21	21.00	0.48	26.70	12.20	33.00	300.00	40.80	1190.00
8.10	0.15	16.20	0.26	22.30	0.70	27.90	31.20	34.70	457.00	42.30	1400.00
9.40	0.16	17.60	0.30	23.30	1.08	28.90	56.00	36.00	580.00		
					[1,1-dimethylurea] $= 0.4$ mol·dm^{-3}						
6.00	0.14	12.80	0.24	18.90	0.84	23.50	27.00	27.80	205.00	32.00	565.00
6.90	0.15	13.30	0.26	20.20	1.80	24.40	48.00	28.90	290.00	33.10	690.00
7.80	0.16	14.90	0.31	21.50	5.10	25.50	88.00	29.70	350.00	34.20	820.00
9.60	0.18	16.40	0.39	22.50	12.20	26.70	150.00	30.80	445.00	35.00	920.00
11.40	0.21	17.90	0.56								

Figure 1. Influence of temperature upon the conductivity of sodium bis(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions in the presence of different ureas and thioureas ([AOT] = 0.5 mol·dm^{-3}, [H$_2$O]/[AOT] = 22), (●) without additive, (▲) [MU] = 0.2 mol·dm^{-3}, (▼) [1,3-DMU] = 0.2 mol·dm^{-3}, (○) [TU] = 0.2 mol·dm^{-3}, (△) [MTU] = 0.2 mol·dm^{-3}, (□) [DMTU] = 0.2 mol·dm^{-3} (concentration in water pool).

Figure 2. Influence of temperature upon the conductivity of sodium bis(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions in the presence of different concentrations of DMTU ([AOT] = 0.5 mol·dm^{-3}, [H$_2$O]/[AOT] = 22): (●) without additive, (▽) [DMTU] = 0.04 mol·dm^{-3}, (□) [DMTU] = 0.2 mol·dm^{-3} (concentration in water pool).

The concentrations of ureas and thioureas considered in this work were less than 1 mol·dm^{-3}. In all the cases the additive concentration has been referred to the volume of the water droplets.

The electrical conductivity was measured employing a conductivimeter Radiometer CDM 3 with a conductivity cell with a constant of 1 cm^{-1}. During the measurements of conductivity the temperature was regulated with a precision of ±0.1 °C. In general, each conductivity value reported was an average of 5 to 10 measurements, where the maximum deviations from the average value were always less than 1.5%. The accuracy of these measurements was ±0.5%. The percolation temperature was determined through the study of the influence of the temperature on the electrical conductivity of the microemulsions.

Results and Discussion

Figure 1 shows the effect of ureas and thioureas on the electrical conductivity of microemulsiones of sodium bis-(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water. In this figure it can be observed how the temperature of electrical percolation decreases significantly upon increasing the degree of methylation of the thiourea (the temperature of percolation can have a reduction of 13 °C). Thus, moderate additive concentrations (ca. 0.2

mol·dm^{-3} referred to the water volume) produce an important decrease in the percolation temperature. On the other hand, Figure 2 shows the effect of increasing the additive concentration on the temperature dependence of the electrical conductivity. In this case we can observe how as the additive concentration is increased an important decrease in the temperature of percolation is produced; thus, in the case of the urea for a concentration of 0.234 mol·dm^{-3} referred to the volume of the disperse phase, a decrease of the temperature of percolation of 22 °C is obtained. A similar behavior was found for microemulsions composed by ternary systems sodium bis(2-ethylhexyl)sulfosuccinate + heptane + water (Costa-Amaral et al., 1992), though much higher concentrations of additive are necessary in order obtain a comparable effect to the one found for sodium bis(2-ethylhexyl)sulfosuccinate + 2,2,4-trimethylpentane + water systems. This smaller effect found for the microemulsions with heptane as continuous phase is primarily due to the lower importance of the interactions between droplets—that produce the phenomenon of the percolation—due to the fact that the chain length of the alkane that forms the continuous phase is smaller.

The values of electrical conductivity/temperature, κ/t, obtained for different additive concentrations are shown in Table 1. From these data it is possible to obtain the temperature at which the percolation process is produced. The percolation threshold has been determined from the
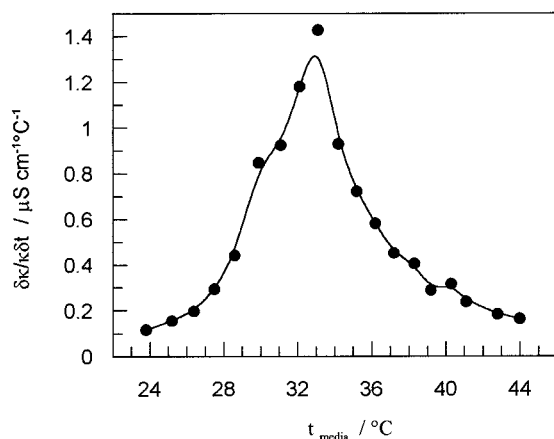
Figure 3. Determination of percolation temperature obtained by Kim method (Kim and Huang, 1986), for sodium bis(2-ethylhexyl)-sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions without additive ([AOT] = 0.5 mol·dm^{-3}, [H$_2$O]/[AOT] = 22).

Table 2. Fitting Parameters (Eq 1) and Percolation Temperature, t_p, Obtained by the Kim Method (Kim and Huang, 1986), for Sodium Bis(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-Trimethylpentane + Water Microemulsions ([AOT] = 0.5 mol·dm^{-3}, [H$_2$O]/[AOT] = 22)

additive	C/mol·dm^{-3}	A	B	C	t_p
none		32.60	0.39	−3.30	33
thiourea	0.040	30.60	0.48	−2.46	31
thiourea	0.200	24.40	0.47	−1.80	25
methylthiourea	0.200	22.80	0.46	−2.20	24
1,3-dimethylthiourea	0.040	29.50	0.46	−2.42	31
1,3-dimethylthiourea	0.200	19.20	0.48	−1.55	20
tetramethylthiourea	0.040	30.80	0.48	−2.66	32
tetramethylthiourea	0.200	22.30	0.51	−2.30	23
urea	0.156	18.00	0.53	−1.82	18
urea	0.234	10.60	0.56	−1.02	11
methylurea	0.200	26.80	0.71	−2.36	27
methylurea	0.400	18.60	0.48	−1.68	19
methylurea	0.810	5.60	0.54	−0.75	6
1,3-dimethylurea	0.200	21.60	0.42	−2.18	22
1,3-dimethylurea	0.400	14.50	0.44	−1.50	15
1,1-dimethylurea	0.200	26.00	0.42	−2.68	26
1,1-dimethylurea	0.400	21.30	0.45	−2.16	21
tetramethylurea	0.200	20.60	0.42	−1.96	21

data κ/t using the treatment illustrated in Figure 3, where the graphics dκ/κdt versus t show a maximum that corresponds with the percolation temperature, t_p (Kim and Huang, 1986). In Table 2 are shown different values for the percolation temperatures in the presence of different additives and of different concentrations in similar conditions ([AOT] = 0.5 mol·dm^{-3}, W = [H$_2$O]/[AOT] = 22). The effect of the thioureas is remarkable, as they turn out to be much more effective in advancing the process of percolation than their oxygenated analogue. The effect exercised by these systems would be justified by their capacity of favoring the opening of channels to facilitate the mass transfer between droplets. In this manner the observed sequence, in which increasing the degree of methylation of the urea or thiourea provokes a greater effect on the moment in which the percolation is produced, would correspond with a decrease of the polarity of the additive, which facilitates a greater association to the film of surfactant. The anomalous results observed for the case of the tetramethylurea would be due to the fact that it is relatively soluble in 2,2,4-trimethylpentane, which would cause this additive to be distributed between the continuous phase and the disperse phase, reducing its efficiency (García-Rio et al., 1994).
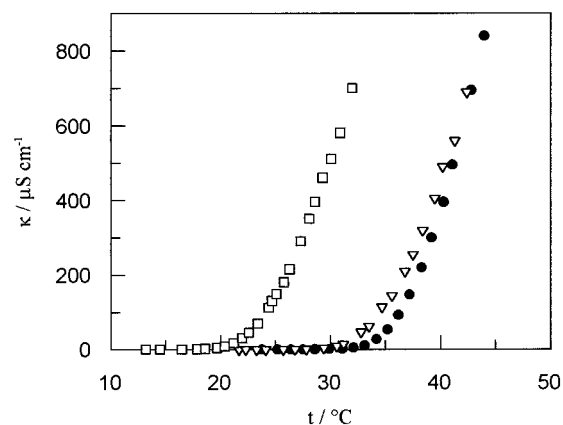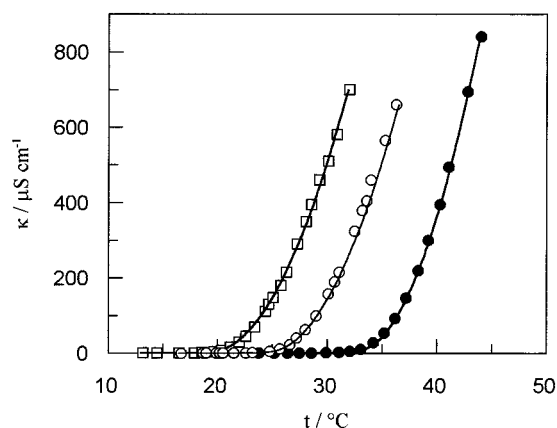

Figure 4. Fit of temperature−conductivity of sodium bis(2-ethylhexyl)sulfosuccinate (AOT) + 2,2,4-trimethylpentane + water microemulsions to eq 1 in the presence of different additive concentrations ([AOT] = 0.5 mol·dm^{-3}, [H$_2$O]/[AOT] = 22): (−) calculated from eq 1, (●) without additive, (○) [TU] = 0.2 mol·dm^{-3}, (□) [DMTU] = 0.2 mol·dm^{-3} (concentration in water pool).

On the other hand, the variation of the conductivity in these systems can be rationalized through the use of an empirical equation that permits us to model the values κ/t before as well as after that the percolation process is produced.

$$t = A + B\sqrt{\kappa} + \frac{C}{\kappa} \qquad (1)$$

The fit of κ/t values was satisfactory in all the cases studied, and the parameters A, B, and C are shown in Table 2. The values of the parameter A (that in all the cases is slightly less than to the value of the percolation threshold obtained by the bibliographical method, vide supra) will correspond with the moment in which the process of percolation is produced.

This equation, where A corresponds to the temperature of percolation, reproduces the experimental data with a deviation less than 4% (see Figure 4).

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