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COMPOUNDING TECHNIQUES FOR ABSORBENT DECONTAMINANTS

Third Quarterly Progress Report
(March 1971 - May 1971)

by

Dewey P. Parks and Robert L. Copeland

June 1971

DEPARTMENT OF THE ARMY
EDGEWOOD ARSENAL
Defense Development and Engineering Laboratories
Physical Protection Laboratory
Edgewood Arsenal, Maryland 21010

Contract Number DAAA15-70-C-0362

Brunswick Corporation
Technical Products Division
Marion, Virginia
24354

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13. ABSTRACT

A study of parameters affecting the spraying of aqueous slurries of active carbon onto surfaces in order to decontaminate same. These slurries incorporate active carbon (minimum of 25%), either coal, petroleum, wood or coconut shell based, a thixotroping agent, a dispersant, a pigment and corrosion inhibitor. Work reported in this report relates to the evaluation of a basic formulation with different types of mixing equipment. A homogenizer type mixer was selected to do additional evaluation with the ball milled slurries for comparative data.

14. Key Words

Activated Carbon	Homogenizer
Dispersants	Dissolver
Thixotropes	High Shear
Pigments	Slurries
Viscosities	Corrosion Inhibitor
Stability	Mixing

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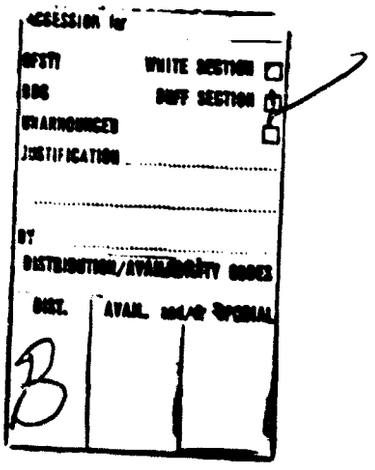

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DEPARTMENT OF THE ARMY
EDGEWOOD ARSENAL
Defense Development and Engineering Laboratories
Physical Protection Laboratory
Edgewood Arsenal, Maryland 21010

Contract Number DAAA15-70-C-0362
Project No. 233-DP-0(RFP-0285)

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Marion, Virginia
24354

SUMMARY

A study was made of the parameters affecting the formulation and application of an aqueous suspension of activated carbon to surfaces in order to decontaminate same. This approach involves combining of activated carbon, a thixotrope, dispersant, corrosion inhibitor, pigment, antifreeze and carrier into a sprayable slurry. A petroleum based carbon was found to be superior to the coal, coconut and wood based carbon in that it provided a stable, sprayable, olive green slurry.

Spraying and adhesion test were successfully completed to prove this particular suspension satisfactory.

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I. INTRODUCTION AND BACKGROUND

The objective of this work is to develop a universal decontaminating system for persistent chemical agents using micronized activated carbon. The decontaminating system must be effective against persistent chemical agents and is designed to the following goals.

1. Act rapidly over a wide temperature range.
2. Be non-toxic.
3. Not damage material and equipment on contact.
4. Be easily and rapidly applied to vehicular equipment, clothing and ancillary equipment.

This program is conducted in three phases, i.e., (A) vehicular decontaminants in the form of a sprayable suspension, (B) clothing and individual equipment decontaminants in the form of dry powders, and (C) scale-up methods and equipment for large volume manufacture and dispensing.

This quarterly report pertains exclusively to the aqueous suspensions for spray application for vehicular and related equipment decontamination.

II. DISCUSSION

A. Objective of Phase I

Phase I of the program involves the formulation of an activated carbon aqueous suspension that will give maximum vapor absorption when sprayed onto a surface. This suspension was formulated from a carbon, thixotrope, dispersant, pigment, corrosion inhibitor, alcohol and water.

Primary objectives are to (1) formulate a suspension that will give maximum stability during storage for two years, (2) can be easily poured and applied by spraying to give a coating of 3-4 mg/sq. cm. of dry active carbon, with sufficient adhesion to resist some degree of abrasion while retaining the maximum absorptive power.

B. Establishment of Basic Materials, Equipment and Methods of Investigation

Work reported in the first and second quarterly progress reports details how the following selections were made of materials, equipment, and methods of investigation

1. Activated Carbons - Wood, coal, petroleum coke, and coconut shell based carbons were evaluated. The average particle size of each of the carbons was 1-2 microns.

Carbon	Manufacturer
CWS - Coal Based	Pittsburgh Activated Carbon Company
Darco KB - Wood Based	Atlas Chemical Company
Type ACC - Petroleum Coke Based	Union Carbide Company
Type UU - Coconut Shell Based	Barnebey-Cheney

2. Thixotropic Agents - Thixotropes were selected on the basis of non-film forming properties and efficiency as thickening agents.

Type	Trade Name/Manufacturer
Montmorillonite (Bentonite Clay)	Bentonite/Fisher Scientific
Resin Grade Asbestos	RG-244/Union Carbide
Pyrogenic (Fumed) Silica	Aerosil-COK 84/DeGussa, Inc.
Microcrystalline Nylon	Aviamide-6P/FMC Corporation

3. Dispersants - Anionic dispersing agents were used to effectively separate the carbon particles for stability.

Type	Trade Name/Manufacturer
Polymerized Salt of Alkyl Naphthalene Sulfonic Acid	DAXAD 11/W. R. Grace Company
Partially Desulfonated Sodium Liquosulfonate	Marasperse CB/American Can
Sodium Salts of Polymerized Substituted Benzoid Alkyl Sulfonic Acid	Darvan No. 2/R. T. Vanderbilt
Water Miscible Polymer	Nopcosant K/Nopco Chemical

4. Pigments - To give an olive green, the following solid pigments were evaluated.

Type	Trade Name/Manufacturer
Iron Oxide	Mapico 1100 Yellow/Columbian Carbon Co.
Phthalocyanine Green	Color No. W-6012/Harshaw Chemical Co.
Cadmium Yellow	Color No. 1489/Harshaw Chemical Co.
Cadmium Yellow	Color No. AZO W-1215/Harshaw Chemical Co.
Cadmium Orange	Color No. 1510/Harshaw Chemical Co.

5. Corrosion Inhibitor - To inhibit rust, the following chemicals were evaluated.

Type	Source
Sodium Chromate	Fisher Scientific
Sodium Silicate	Fisher Scientific

6. Solvent/Antifreeze - As a carrier and antifreeze, the following was used.

Water - Demineralized	60 parts by volume
Methyl Alcohol	40 parts by volume

7. Mixing Equipment - After evaluating three different mixing techniques, i.e., the ball mill, homogenizer and Cowles dissolver; the homogenizer type mixer was found to be superior from the standpoint of uniformity and ease of mixing. The detailed evaluation is described in the first and second quarterly progress reports.

8. Methods of Evaluation and Investigation - Due to the infinite number of possible combinations of materials and mixing equipment, key combinations of materials were selected and evaluated based on uniformity, stability, color, ease of mixing and viscosities. It is the intention to first establish the most efficient thixotrope based on a high viscosity; second, establish the most effective dispersant based on a low viscosity; third, add active carbon until a workable viscosity is formed, and fourth, determine the optimum percent concentration of dispersant by measuring the viscosity and observing the stability at 1, 2, 3, and 4 percent concentration. Generally, materials were eliminated from the program based upon their performance when put into suspension.

C. Evaluation of Each of the Different Based Carbons in Suspension

1. CWS Carbon - Coal Based

The coal based activated carbon used in this evaluation was manufactured by the Pittsburgh Activated Carbon Company. The carbon supplied to Brunswick by Edgewood Arsenal had an average particle size of 1.5 μ and a pH value of 10.0 in aqueous solution. The surface area measured 1000-1100 sq. meters/gram with a CCL_4 activity of 65%.

The different thixotropic and dispensing agents, shown in Section B, were first evaluated for effects on aqueous slurries of CWS activated carbon. Formulations were prepared with each thixotrope, i.e., bentonite, asbestos, pyrogenic silica and microcrystalline nylon, and are shown in Table I.

Experiments 79, 83, 87, and 87-A show effects of each of the thixotropes when using Darvan No. 1 as the dispersing agent. As this series of experiments show, bentonite was the most efficient thixotropic agent in

that it gave the highest, most stable viscosity. Experiments 115, 116, 117 and 118 were performed under the same condition substituting DAXAD 11 as the dispersant. Results again show bentonite to be the most efficient thixotropic agent by giving the highest viscosity with the other additives being held constant.

It was concluded from these two series of experiments that bentonite is a superior thixotropic agent to either asbestos, pyrogenic silica or microcrystalline nylon when using the CWS coal based carbon.

After selecting bentonite as the thixtrope agent for CWS carbon, different dispersing agents, DAXAD 11, Darvan No. 2, Marasperse CB, and Nopcosant K were evaluated for their effectiveness in lowering the viscosity. Experiments 115, 119, 120, and 121 (Table II) show initial evaluations of the dispersants. By assuming the lower viscosity indicates a better dispersion of carbon particles, experiment 120, using Marasperse CB dispersant, was selected for additional evaluation.

By analyzing experiments 134, 135, 137, and 141 (Table III) it can be seen that carbon loadings up to 40% (Exp. 137) are possible using Marasperse CB, however, these slurries become glue-like in texture with a significant drop in viscosity during storage. Experiments 144, 145, and 147 were conducted to maximize carbon loading with a more stable viscosity. Experiment 145 using DAXAD 11 gave both a carbon content of 31.7% and more stable viscosity during storage. Therefore, DAXAD 11 was chosen as the superior dispersant.

After selecting bentonite as the most efficient thixotrope and DAXAD 11 as the dispersing agent, the next step was to determine the optimum percent concentrations of DAXAD 11. This was done by conducting the experiments shown in Table IV. The results are also shown charted in Figure 1. Experiments 115, 149, 150, and 151 were conducted with a 300 gram CWS carbon loading ($\sim$ 25%) with 12, 24, 36, and 48 grams ($\sim$ 1, 2, 3, & 4%) of DAXAD 11 dispersant.

Experiments 152, 145, 153, and 154 contained 395 grams (31%) carbon loading. As the results show, there is a significant difference in reaction of the DAXAD 11 to the percent carbon loadings. This data indicates optimum to be 2% DAXAD 11 dispersant with each carbon loading.

The next series of experiments were prepared to test the effect of corrosion inhibitors. Experiments 145, 172, and 176, shown in Table V, give the resulting viscosities of suspension prepared with sodium chromate and sodium silicate inhibitors. Both Experiments 176 and 172, containing .48% and .96% sodium silicate inhibitor, show a drop in viscosity with eventual settling. Sodium silicate did show a slight increase in pH over the sodium chromate. Based on stability of the suspensions, sodium chromate is selected as the better corrosion inhibitor.

Experiments 145, 184, 185, 186, and 201, shown in Table VI, were conducted to evaluate the effect of various mixing times using the L-1 Laboratory Homogenizer. As the results show, each increased mixing time produced a higher viscosity, however no apparent stabilization of the viscosities was achieved. It was concluded that additional mixing time beyond thirty (30) minutes does not improve the stability of the CWS suspensions. The solvent content of Experiments 145 and 201 measured 55.8% and 55.5% respectively, therefore solvent evaporation during mixing did not produce the increased viscosities.

Various types of pigments were evaluated to produce an olive green color. Cadmium lithopone yellows, oranges and phthalocyanine green pigments supplied by Harshaw Chemical Company were added to CWS suspension in concentrations up to 6.3%. As Table VII shows, Experiments 191 with the Azo yellow and 193 with the cadmium lithopone gave considerable increases in viscosity without the desired olive green color. A 6.3% concentration of phthalocyanine green remained black. The Mapico 1100 yellow, iron oxide, pigment did give the desired olive green at a 4.8% level, therefore was selected over the other pigments tested.

After making final selections of a thixotrope, dispersant, pigment and corrosion inhibitor for use with the CWS carbon, some adjustments were made in solvent content. With a carbon content of 31.60% and a solvent content of 60.71% the resulting viscosity was in the 25,000 centipoise range. As can be seen by Spray Test No. 12, the coverage over the test panel was in the 20 mg/cm^2 range indicating the viscosity was too high for effective spraying. Also the resulting spray gave a spattered, non-uniform coating again indicating the material to be thick. The solvent content was then increased to 63.03% (reducing the carbon to 29.59%) which resulted in a viscosity in the 12-14,000 centipoise range. Slurries in this viscosity range can be effectively sprayed.

A typical formulation for CWS carbon used both for testing sprayability and adhesion would be as follows:

Ingredient	Actual Weight	Percent Formula Wt.
Carbon: CWS Carbon	385 g	29.59
Thixotrope: Bentonite	6 g	.46
Dispersant: DAXAD 11	24 g	1.84
Pigment: 1100 Yellow	60 g	4.61
Corrosion Inhibitor: Sodium Chromate	6 g	.46
Solvent: (60/40 Water-Methanol)	820 ml	63.03
Mixing Time: Thirty (30) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours after mixing	14,000 cps Helipath 11,000 cps #4 @ 10 RPM 5,500 cps #4 @ 20 RPM	
pH Value	10.4	
Carbon Particle Size	1-2 μ (avg.)	

TABLE I

CARBON SUSPENSIONS

CWS CARBON - THIXOTROPE EVALUATION

FORMULATION NO.	79	83	87	87-A	115	116	117	118
COMPOSITION BY/WT.	Actual Wt.							
Carbon CWS	300 g	300 g	300 g	300 g	300 g	300 g	300 g	300 g
Thixotrope (1) Bentonite	6 g				6 g			
(2) RG-244		6 g				6 g		
(3) COK-84			6 g				6 g	
(4) Nylon-6				6 g				6 g
Dispersant (1) DAXAD 11					12 g	12 g	12 g	12 g
(2) Darvan #1	12 g	12 g	12 g	12 g				
(3) Marasperse CB								
(4) Nopcosant K								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_1O_4$	6 g	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2) $Na_2S_1O_3$								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.	60	60	60	60	30	30	30	30
CHARACTERISTICS								
Viscosity, CP								
Imed. After Mix Helipath "C"	9,000	2,000	3,000	1,000	7,000	1,800	3,000	1,500
Imed. After Mix #4 @ 10 RPM	6,800	1,900	2,800	500	5,700	1,100	2,440	1,000
Imed. After Mix #4 @ 20 RPM	3,400	1,050	1,500	300	2,875	575	1,325	500
After 24 Hours Helipath "C"	12,000	2,800	4,000	500	10,000	2,000	4,000	2,500
After 24 Hours #4 @ 10 RPM	9,000	2,200	3,600	200	7,900	1,200	3,350	1,900
After 24 Hours #4 @ 20 RPM	4,700	1,100	1,900	150	4,000	600	1,800	950
After 7 Days Helipath "C"	16,000	3,600	6,700		14,000	2,500	6,500	3,000
After 7 Days #4 @ 10 RPM	12,000	2,900	5,600		13,700	1,600	5,000	2,100
After 7 Days #4 @ 20 RPM	6,200	1,300	2,750		4,850	775	2,650	1,000
After 14 Days Helipath "C"	17,000	2,000	7,000	Discarded - Too Thin	14,000	2,900	6,800	2,500
After 14 Days #4 @ 10 RPM	12,500	1,400	5,700		10,200	1,500	5,350	2,300
After 14 Days #4 @ 20 RPM	8,150	750	2,800		5,200	800	2,900	1,175
After 30 Days Helipath "C"	16,800	3,700	8,000		14,000	2,000	7,000	2,900
After 30 Days #4 @ 10 RPM	13,000	2,100	5,800		11,300	1,100	5,000	2,150
After 30 Days #4 @ 20 RPM	6,800	1,125	2,800		5,550	600	2,550	1,000
pH	10.5	10.3	10.2		9.7	9.5	9.9	9.8

NOTES:

TABLE II

CARBON SUSPENSIONS
CWS CARBON - DISPERSANT EVALUATION

FORMULATION NO.		115	119	120	121			
COMPOSITION BY/WT.				Actual Wt.				
Carbon	CWS	300 g	300 g	300* g	300 g			
Thixotrope (1)	Bentonite	6 g	6 g	6 g	.6 g			
(2)								
(3)								
(4)								
Dispersant (1)	DAXAD 11	12 g						
(2)	Darvan No. 2		12 g					
(3)	Marasperse CB			12 g				
(4)	Nopcosant K				12 g			
Pigment	1100 Yellow	60 g	60 g	60 g	60 g			
Corrosive Inhibitor (1)	$Na_2C_2O_4$	6 g	6 g	6 g	6 g			
(2)								
Solvent, Water/Alcohol 60/40 By Vol.		756 ML	756 ML	756 ML	756 ML			
MIXING TECHNIQUE : Homogenizer, min.		30	30	30	30			
CHARACTERISTICS								
Viscosity, CP								
Imed. After Mix	Helipath "C"	7,000	3,500	1,100	11,000			
	#4 @ 10 RPM	5,700	2,200	650	9,600			
	#4 @ 20 RPM	2,875	1,225	400	5,675			
After 24 Hours	Helipath "C"	10,000	1,500	1,000	9,400			
	#4 @ 10 RPM	7,900	900	600	8,500			
	#4 @ 20 RPM	4,000	500	350	4,550			
After 7 Days	Helipath "C"	14,000	1,000	1,800	7,500			
	#4 @ 10 RPM	13,700	450	1,000	7,250			
	#4 @ 20 RPM	4,850	300	550	3,875			
After 14 Days	Helipath "C"	14,000	1,000	1,800	7,300			
	#4 @ 10 RPM	10,200	400	1,000	6,800			
	#4 @ 20 RPM	5,200	250	550	3,675			
After 30 Days	Helipath "C"	14,000		1,500	6,800			
	#4 @ 10 RPM	11,300	Discarded Too Thin	750	6,000			
	#4 @ 20 RPM	5,550		425	3,325			
pH		9.7	9.5	9.6	9.8			

NOTES: *MOST EFFICIENT DISPERSANT

TABLE III

CARBON SUSPENSIONS
CWS CARBON - DISPERSANT EVALUATION

FORMULATION NO.		134	135	137	141	144	145	147
COMPOSITION BY/WT.				Actual Wt.				
Carbon	CWS	425 g	450 g	500*g	450 g	430 g	395 g	325 g
Thixotrope (1)	Bentonite	6 g	6 g	6 g	9 g	6 g	6 g	6 g
(2)								
(3)								
(4)								
Dispersant (1)	Maresperse CB	12+12 g	24 g	24 g	24 g			
(2)	Darvan No. 2					24 g		
(3)	DAXAD 11						24 g	
(4)	Nopcosant K							24 g
Pigment	1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1)	$Na_2C_2O_4$	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2)								
Solvent, Water/Alcohol 60/40 By Vol.		756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.		30+30	30	30	30	30	30	30
CHARACTERISTICS								
Viscosity, CP		(34,000)						
Imed. After Mix	Helipath "C"	10,000	6,000	15,000	15,000	14,500	16,500	11,000
	#4 @ 10 RPM	5,400	2,800	6,300	6,000	10,250	13,650	10,300
	#4 @ 20 RPM	3,350	1,900	4,750	3,950	6,350	6,650	5,450
After 24 Hours	Helipath "C"	5,000	2,000	12,000	7,000	6,000	16,000	10,000
	#4 @ 10 RPM	1,850	1,100	4,900	2,900	3,725	13,000	9,200
	#4 @ 20 RPM	1,200	775	3,650	1,950	2,500	6,325	4,900
After 7 Days	Helipath "C"	3,000	1,500	8,000	5,000	3,200	23,500	9,000
	#4 @ 10 RPM	1,300	900	2,900	2,100	1,900	17,600	8,200
	#4 @ 20 RPM	900	700	2,225	1,400	1,300	8,650	4,500
After 14 Days	Helipath "C"	2,000	1,200	5,000	4,000	2,800	25,000	8,800
	#4 @ 10 RPM	1,000	800	2,200	1,625	1,400	19,600	7,700
	#4 @ 20 RPM	675	600	1,700	1,125	950	9,800	4,200
After 30 Days	Helipath "C"	1,000	900	3,000	2,000	2,000	28,000	8,000
	#4 @ 10 RPM	700	600	2,000	1,150	1,200	22,000	6,300
	#4 @ 20 RPM	500	450	1,650	850	850	11,100	3,500
pH		10.1	10.6	10.1	10.2	9.6	10.0	10.0

NOTES: *MIXTURE BECAME GLUE-LIKE WITH 500 g CARBON

TABLE IV

CARBON SUSPENSIONS
CWS CARBON - PERCENT DISPERSANT EVALUATION

FORMULATION NO.	115	149	150	151	152	145	153	154
COMPOSITION BY/WT.			Actual Wt.					
Carbon CWS	300 g	300 g	300 g	300 g	395 g	395 g	395 g	395 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2)								
(3)								
(4)								
Dispersant (1) DAXAD 11	12 g	24 g	36 g	48 g	12 g	24 g	36 g	48 g
(2)								
(3)								
(4)								
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_4O_4$	6 g	6 g	6 g	6 g	6 g	6 g	6 g	6 g
(2)								
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30	30	30	30
CHARACTERISTICS								
Viscosity, CP								
Imed. After Mix Helipath "C"	7,000	4,000	5,000	5,500	25,000	16,500	18,000	26,000
Imed. After Mix #4 @ 10 RPM	5,700	3,000	3,600	3,850	22,900	13,650	13,200	19,800
Imed. After Mix #4 @ 20 RPM	2,875	1,575	1,750	1,875	11,450	6,650	6,850	9,700
After 24 Hours Helipath "C"	10,000	6,000	6,000	5,900	28,000	16,000	21,500	24,000
After 24 Hours #4 @ 10 RPM	7,900	4,150	4,200	4,300	26,400	13,000	16,400	17,400
After 24 Hours #4 @ 20 RPM	4,000	2,050	2,100	2,140	13,800	6,325	8,100	8,500
After 7 Days Helipath "C"								
After 7 Days #4 @ 10 RPM								
After 7 Days #4 @ 20 RPM								
After 14 Days Helipath "C"								
After 14 Days #4 @ 10 RPM								
After 14 Days #4 @ 20 RPM								
After 30 Days Helipath "C"								
After 30 Days #4 @ 10 RPM								
After 30 Days #4 @ 20 RPM								
pH								

NOTES:

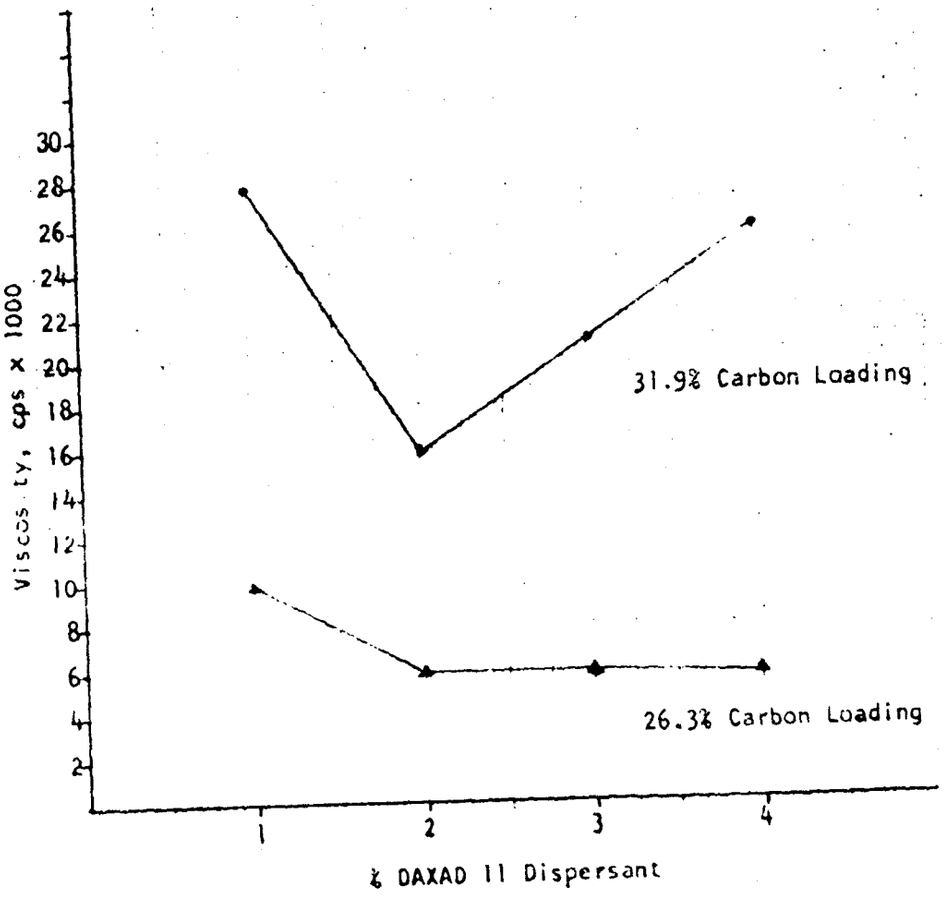

FIGURE 1 - EFFECT OF % DISPERSANT ON BPL(CWS) CARBON SLURRIES

TABLE V

CARBON SUSPENSIONS
CWS CARBON - CORROSION INHIBITOR

FORMULATION NO.	145	176	172
COMPOSITION BY/WT.			Actual Wt.
Carbon CWS	395 g	395 g	395 g
Thixotrope (1) Bentonite	6 g	6 g	6 g
(2)			
(3)			
(4)			
Dispersant (1) DAXAD 11	24 g	24 g	24 g
(2)			
(3)			
(4)			
Pigment 1100 Yellow	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_1O_4$	6 g		
(2) $Na_2S_1O_3$		6 g	12 g
Solvent, Water/Alcohol 60/40 By Vol.	756 ml	756 ml	756 ml
MIXING TECHNIQUE Homogenizer, minutes	30	30	30
CHARACTERISTICS			
Viscosity, CP			
Imed. After Mix — Helipath "C"	16,500	4,000	11,000
Imed. After Mix — #4 @ 10 RPM	13,650	2,600	6,500
Imed. After Mix — #4 @ 20 RPM	6,650	1,600	3,425
After 24 Hours — Helipath "C"	16,000	1,000	1,000
After 24 Hours — #4 @ 10 RPM	13,000	1,100	1,900
After 24 Hours — #4 @ 20 RPM	6,325	700	1,100
After 7 Days — Helipath "C"	23,500	< 500	< 500
After 7 Days — #4 @ 10 RPM	17,600	~ 300	~ 300
After 7 Days — #4 @ 20 RPM	8,650	~ 250	~ 250
After 14 Days — Helipath "C"	25,000		
After 14 Days — #4 @ 10 RPM	19,600		
After 14 Days — #4 @ 20 RPM	9,800	Thin Settling	Very Thin Settling
After 30 Days — Helipath "C"	28,000		
After 30 Days — #4 @ 10 RPM	22,000		
After 30 Days — #4 @ 20 RPM	11,100		
pH	10.0	10.7	10.8

NOTES:

TABLE VI

CARBON SUSPENSIONS
CWS CARBON - MIXING TIME STUDY

FORMULATION NO.		145	184	185	188	201
COMPOSITION BY/WT.				Actual Wt.		
Carbon CWS		395 g	395 g	395 g	395 g	395 g
Thixotrope (1) Bentonite						
(2)						
(3)						
(4)						
Dispersant (1) DAXAD 11		24 g	24 g	24 g	24 g	24 g
(2)						
(3)						
(4)						
Pigment 1100 Yellow		60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$		6 g	6 g	6 g	6 g	6 g
(2)						
Solvent, Water/Alcohol 60/40 By Vol., ml		756	756	756	756	756
MIXING TECHNIQUE: Homogenizer, minutes		30	60	90	120	180
CHARACTERISTICS						
Viscosity, CP						
Imed. After Mix	Helipath "C"	16,500	19,000	20,000	28,000	45,000
	#4 @ 10 RPM	13,650	14,300	15,300	20,200	28,000
	#4 @ 20 RPM	6,650	7,100	7,625	9,900	14,400
After 24 Hours	Helipath "C"	16,000	29,000	28,000	27,000	48,000
	#4 @ 10 RPM	13,000	19,800	18,000	19,000	32,200
	#4 @ 20 RPM	6,325	9,700	9,100	9,650	16,400
After 7 Days	Helipath "C"	23,500	34,000	32,000	41,000	53,000
	#4 @ 10 RPM	17,600	25,100	23,200	29,400	35,600
	#4 @ 20 RPM	8,650	12,500	12,000	15,100	18,000
After 14 Days	Helipath "C"	25,000	36,000	34,000	42,000	60,000
	#4 @ 10 RPM	19,600	26,200	25,000	33,200	32,500
	#4 @ 20 RPM	9,800	12,800	12,200	16,800	19,000
After 30 Days	Helipath "C"	28,000	34,000	34,000	45,000	57,000
	#4 @ 10 RPM	22,000	26,400	25,600	33,200	37,500
	#4 @ 20 RPM	11,100	13,700	13,500	17,500	18,000
pH		10.0	10.0	10.1	10.0	10.0
NOTES: Solvent Content, %		55.8				55.5

TABLE VII

CARBON SUSPENSIONS
CWS CARBON - PIGMENT EVALUATION

FORMULATION NO.	145	191	192	193
COMPOSITION BY/WT.			Actual Wt.	
Carbon CWS	395 g	395 g	395 g	395 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g
(2)				
(3)				
(4)				
Pigment (1) 1100 Yellow	60 g			
(2) Azo Yellow		80 g		
(3) Phthalocyanine Green*			80 g	
(4) Cad Yellow/Cad Orange				56/5 g
Dispersant DAXAD 11	24 g	24 g	24 g	24 g
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	6 g	6 g	6 g
(2)				
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756
MIXING TECHNIQUE	30	30	30	30
CHARACTERISTICS				
Viscosity, CP				
Imed. After Mix Helipath "C"	16,500	42,000	3,500	46,000
Imed. After Mix #4 @ 10 RPM	13,600	20,400	1,200	29,800
Imed. After Mix #4 @ 20 RPM	6,650	10,300	750	16,200
After 24 Hours Helipath "C"	16,000			
After 24 Hours #4 @ 10 RPM	13,000			
After 24 Hours #4 @ 20 RPM	6,325			
Color, Wet	Olive Green	Dark Green	Black	Medium Green
Color, Dry	Olive Green	Green Black	Black	Dark Green
pH				

NOTES: *Supplied by Harshaw Chemical in aqueous suspension

2. Darco KB Carbon - Wood Based

The wood based carbon is manufactured by the Atlas Chemical Company and is supplied with an average particle size of 1.2 μ. The pH value in aqueous solution is approximately 5.2 with a surface area of 600-700 square meters/gram and a CCL_4 activity of 84%.

In evaluating Darco KB carbon, the same general plan of experimentation was used as with the CWS carbon. Experiments 95, 96, 97, 103, 104, and 106, shown on Table VIII, give results when the carbon is combined with each of the thixotropic agents. After solvent adjustment was made (Exp. 95, 96, and 97) to keep the slurry sufficiently fluid to mix, slurries were prepared with each of the thixotropes using DAXAD 11 as the dispersant. By analyzing Experiments 97, 103, 104, and 106, it can be concluded that bentonite was the most efficient thixotropic agent in that it gave the highest workable viscosity. Although RG-244 asbestos, shown in Experiment 103, gave a very high viscosity, in order to prepare a mixable slurry it would be necessary to (1) lower the carbon content below 24%, (2) lower the thixotrope below 1/2%, which would not be a sufficient amount to hold the carbon particles in suspension, or (3) raise the solvent content, all of which are undesirable.

It was concluded from this series of experiments that bentonite is the superior thixotropic agent with Darco KB wood based carbon.

In evaluating dispersants with the Darco KB-bentonite system, slurries were prepared as shown in Table IX. It can be seen that Darvan No. 2 give the lowest viscosity, indicating the best dispersion of carbon particles. Experiment 148 was conducted using a cationic dispersant, Ammonyx 856, but the data shows the anionic dispersants gave a lower viscosity indicating better dispersion. Darvan No. 2 dispersant was selected for further evaluation.

Table X shows results of increasing the carbon content of the Darco KB-bentonite system and the effect on viscosities. Experiment 143 gave the maximum loading at 25.4% carbon with the lowest viscosity. This was accomplished with the Darvan No. 2 as the dispersant.

Experiment 136 shows the two step procedure used to arrive at the maximum carbon loading.

With the Darco KB carbon, bentonite was selected as the thixotrope and Darvan No. 2 as the dispersant. To determine the optimum percent contentration of dispersant, a series of experiments were made and the results shown in Table XI and Figure II. Unlike the coal based carbon (CWS) suspensions, Darco KB wood based carbon slurries show a sharp drop in viscosity as the percent dispersant is raised to 5%. As shown in Figure II, the viscosity of the 26% carbon loaded slurry was stabilized between 3% and 5% with only a slight decrease in viscosity at the 5% level.

The data indicates the optimum to be 3% of Darvan No. 2 dispersant with the carbon loading of 25.2%.

Table XII gives results of investigating sodium chromate and sodium silicate as the corrosion inhibitor. Although the suspension containing sodium silicate remained very stable during storage, the pH was unacceptably low at 6.1. Sodium chromate was selected as the corrosion inhibitor.

In finalizing the selection of the Darco KB carbon suspension, Experiments 159, 189 and 199, shown in Table XIII, were conducted. After 90 minutes mixing time, the suspensions showed large increases in viscosity with some friction heating being experienced with Experiment No. 199. Again solvent evaporation was not the apparent cause of the viscosity increase. Therefore, it is concluded that a thirty (30) minute mix time produces a uniform suspension with no improvements in stability by additional mixing.

Pigment evaluation shown by Experiments 159, 206, 207, and 208 in Table XIV for the most part produced the same results as with the CWS carbon. Mapico 1100 yellow, iron oxide, gave a suitable olive green at 5.05% while higher concentrations (5.44%) of the cadmium lithopones resulted in color gradients ranging from dark green to black. Again the iron oxide pigment was chosen to give the olive green.

Of the different based carbons formulated and tested, the Darco KB appears to be the least attractive for a spray application. In order to prepare a sprayable slurry it was necessary to adjust the solvent content upward to 67.26% which gave a carbon content of 24.29%. The resulting viscosity measured 24 hours after mixing was 17,000 centipoise. As is characteristic of this particular carbon, the viscosity continues to drop during storage however does appear to stabilize after 30 days.

After formulation adjustments to give a sprayable system a typical formulation used for both testing sprayability and adhesion would be as follows:

Ingredient	Actual Weight	Percent Formula Wt.
Carbon: Darco KB	325 g	24.29
Thixotrope: Bentonite	6 g	.45
Dispersant: Darvan No. 2	36 g	2.69
Pigment: 1100 Yellow	65 g	4.86
Corrosion Inhibitor: Sodium Chromate	6 g	.45
Solvent: (60/40 Water-Methanol)	900 ml	67.26
Mixing Time: Thirty (30) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours after mixing	17,000 cps Helipath 14,800 cps #4 @ 10 RPM 7,450 cps #4 @ 20 RPM	
pH Value	10.4	
Carbon Particle Size	1-2 μ (avg.)	

TABLE VIII

CARBON SUSPENSIONS

DARCO KB CARBON - THIXOTROPES

FORMULATION NO.	95	96	97	103	104	106
COMPOSITION BY/WT.			Actual Wt.			
Carbon DARCO KB	300 g	300 g	300 g	300 g	300 g	300 g
Thixotrope (1) Bentonite	6 g	6 g	6 g			
(2) RG-244				6 g		
(3) COK -84					6 g	
(4) Nylon-6						6 g
Dispersant (1) DAXAD 11	12 g	12 g	12 g	12 g	12 g	12 g
(2) Darvan No. 2						
(3) Marasperse CB						
(4) Nopcosant K						
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) Na_2CrO_4	6 g	6 g	6 g	6 g	6 g	6 g
(2)						
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	956 ML	856 ML	856 ML	856 ML	856 ML
MIXING TECHNIQUE: Homogenizer, min.	--	30	30	--	30	30
CHARACTERISTICS						
Viscosity, CP						
Imed. After Mix — Helipath "C"		10,000	16,000		14,000	12,800
Imed. After Mix — #4 @ 10 RPM	-	8,500	14,700	-	12,500	11,800
Imed. After Mix — #4 @ 20 RPM	Discarded - too viscous to mix after 2 min.	4,200	7,650	Discarded - too viscous to mix after 2 min.	6,300	6,050
After 24 Hours — Helipath "C"		8,000	15,000		11,000	10,000
After 24 Hours — #4 @ 10 RPM		7,000	13,200		10,800	9,300
After 24 Hours — #4 @ 20 RPM		3,700	7,000		5,500	4,075
After 7 Days — Helipath "C"		7,450	13,000		9,000	9,000
After 7 Days — #4 @ 10 RPM		6,300	11,700		8,900	7,700
After 7 Days — #4 @ 20 RPM		3,300	6,175		4,600	3,950
After 14 Days — Helipath "C"	-	7,000	13,000	-	9,000	8,200
After 14 Days — #4 @ 10 RPM		6,200	11,800		8,800	7,625
After 14 Days — #4 @ 20 RPM		3,200	6,300		4,650	3,925
After 30 Days — Helipath "C"	-	8,000	14,800	-	11,000	8,500
After 30 Days — #4 @ 10 RPM		6,800	13,550		10,350	7,625
After 30 Days — #4 @ 20 RPM		3,625	7,200		5,350	4,000
pH		8.8	8.8		8.3	7.7

NOTES:

TABLE IX

CARBON SUSPENSIONS

DARCO KB CARBON - DISPERSANTS

FORMULATION NO.	97	112	113	105		148
COMPOSITION BY/WT.			Actual Wt.			
Carbon DARCO KB	300 g	300 g	300 g	300 g		300 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g		6 g
(2)						
(3)						
(4)						(Ammonyx 856)*
Dispersant (1) DAXAD 11	12 g					12 g
(2) Nopcosant K		12 g				
(3) Marasperse CB			12 g			
(4) Darvan No. 2				12 g		
Pigment 1100 Yellow	60 g	60 g	60 g	60 g		60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	6 g	6 g	6 g		6 g
(2)						
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML		856 ML
MIXING TECHNIQUE : Homogenizer, min.	30	30	30	30		30

CHARACTERISTICS

Viscosity, CP						
Imed. After Mix	Helipath "C"	16,000	30,000	13,000	7,000	41,000
	#4 @ 10 RPM	14,700	26,000	11,540	6,700	12,300
	#4 @ 20 RPM	7,650	13,950	6,000	3,475	12,100
After 24 Hours	Helipath "C"	15,000	19,000	11,000	4,200	32,000
	#4 @ 10 RPM	13,200	18,800	9,400	3,800	12,000
	#4 @ 20 RPM	7,000	9,850	4,950	2,075	11,400
After 7 Days	Helipath "C"	13,000	18,200	7,300	4,000	28,000
	#4 @ 10 RPM	11,700	16,000	6,300	3,100	12,000
	#4 @ 20 RPM	6,175	8,425	3,350	1,700	11,750
After 14 Days	Helipath "C"	13,000	15,000	6,000	3,800	30,000
	#4 @ 10 RPM	11,800	14,600	4,900	3,200	13,000
	#4 @ 20 RPM	6,300	7,700	2,600	1,700	2,650
After 30 Days	Helipath "C"	14,800	16,000	6,900	4,000	30,000
	#4 @ 10 RPM	13,550	15,500	5,600	3,600	13,800
	#4 @ 20 RPM	7,200	8,150	2,950	1,950	12,600
pH		8.8	8.3	7.9	7.8	7.6

NOTES: *CATIONIC DISPERSANT

TABLE X

CARBON SUSPENSIONS
DARCO KB CARBON - % DISPERSANT

FORMULATION NO.	133	136	143	142			
COMPOSITION BY/WT.			Actual Wt.				
Carbon DARCO KB	300 g	325 g	325 g	325 g			
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g			
(2)							
(3)							
(4)							
Dispersant (1) DAXAD 11	24 g						
(2) Darvan No. 2		12 + 12	24 g				
(3) Marasperse CB				24 g			
(4)							
Pigment 1100 Yellow	60 g	60 g	60 g	60 g			
Corrosive Inhibitor (1) $Na_2C_2O_4$	6 g	6 g	6 g	6 g			
(2)							
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML			
MIXING TECHNIQUE: Homogenizer, min.	30	30 + 30	30	30			

CHARACTERISTICS

Viscosity, CP		(33,000)						
Imed. After Mix	Helipath "C"	32,000	25,000	25,000	42,000			
	#4 @ 10 RPM	31,400	24,000	22,700	34,000			
	#4 @ 20 RPM	16,500	12,300	12,200	19,250			
After 24 Hours	Helipath "C"	32,000	21,000	21,000	35,000			
	#4 @ 10 RPM	31,300	20,800	20,800	30,000			
	#4 @ 20 RPM	16,450	10,500	11,000	17,250			
After 7 Days	Helipath "C"	32,000	20,500	19,000	24,500			
	#4 @ 10 RPM	30,600	20,100	18,800	23,600			
	#4 @ 20 RPM	16,050	10,300	9,950	12,400			
After 14 Days	Helipath "C"	34,000	22,000	20,000	24,500			
	#4 @ 10 RPM	33,000	23,600	19,600	24,200			
	#4 @ 20 RPM	17,550	12,000	10,350	12,650			
After 30 Days	Helipath "C"	34,500	25,000	23,000	31,000			
	#4 @ 10 RPM	32,200	26,600	22,600	30,200			
	#4 @ 20 RPM	17,100	13,700	11,900	15,900			
pH		7.7	8.0	7.9	7.9			

NOTES:

TABLE XI

CARBON SUSPENSIONS
DARCO KB CARBON - % DISPERSANT EVALUATION

FORMULATION NO.	148	155	156	158	162		
COMPOSITION BY/WT.			Actual Wt.				
Carbon DARCO KB	300 g	300 g	300 g	300 g	300 g		
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g		
(2)							
(3)							
(4)							
Dispersant (1) Darvan No. 2	12 g	24 g	36 g	48 g	60 g		
(2)							
(3)							
(4)							
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g		
Corrosive Inhibitor (1) $Na_2C_2O_4$	6 g	6 g	6 g	6 g	6 g		
(2)							
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML	856 ML		
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30		
CHARACTERISTICS							
Viscosity, CP							
Imed. After Mix — Helipath "C"	7,000	13,500	10,000	9,000	1,000		
Imed. After Mix — #4 @ 10 RPM	6,700	13,300	8,800	5,100	525		
Imed. After Mix — #4 @ 20 RPM	3,475	6,800	4,500	2,625	350		
After 24 hours — Helipath "C"	4,200	10,500	7,000	5,500	500		
After 24 hours — #4 @ 10 RPM	3,800	10,800	6,500	3,300	300		
After 24 hours — #4 @ 20 RPM	2,075	5,600	3,350	1,750	250		
After 7 Days — Helipath "C"							
After 7 Days — #4 @ 10 RPM							
After 7 Days — #4 @ 20 RPM							
After 14 Days — Helipath "C"							
After 14 Days — #4 @ 10 RPM							
After 14 Days — #4 @ 20 RPM							
After 30 Days — Helipath "C"							
After 30 Days — #4 @ 10 RPM							
After 30 Days — #4 @ 20 RPM							
pH							

NOTES:

TABLE XI (Cont'd)

CARBON SUSPENSIONS
DARCO KB CARBON - % DISPERSANT EVALUATION

FORMULATION NO.	157	143	159	160	163		
COMPOSITION BY/WT.			Actual Wt.				
Carbon DARCO KB	325 g	325 g	325 g	325 g	325 g		
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g		
(2)							
(3)							
(4)							
Dispersant (1) Darvan No. 2	12 g	24 g	36 g	48 g	60 g		
(2)							
(3)							
(4)							
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g		
Corrosive Inhibitor (1) $Na_2C_2O_4$	6 g	6 g	6 g	6 g	6 g		
(2)							
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML	856 ML	856 ML		
MIXING TECHNIQUE : Homogenizer, min.	30	30	30	30	30		
CHARACTERISTICS							
Viscosity, CP							
Immed. After Mix — Helipath "C"	29,000	25,000	14,000	13,500	13,000		
Immed. After Mix — #4 @ 10 RPM	24,800	22,700	11,900	10,400	7,450		
Immed. After Mix — #4 @ 20 RPM	14,800	12,200	5,925	5,325	3,925		
After 24 Hours — Helipath "C"	22,000	21,000	10,000	9,000	7,500		
After 24 Hours — #4 @ 10 RPM	21,500	20,000	8,900	6,800	4,450		
After 24 Hours — #4 @ 20 RPM	11,950	11,000	4,550	3,575	2,500		
After 7 Days — Helipath "C"							
After 7 Days — #4 @ 10 RPM							
After 7 Days — #4 @ 20 RPM							
After 14 Days — Helipath "C"							
After 14 Days — #4 @ 10 RPM							
After 14 Days — #4 @ 20 RPM							
After 30 Days — Helipath "C"							
After 30 Days — #4 @ 10 RPM							
After 30 Days — #4 @ 20 RPM							
pH							

NOTES:

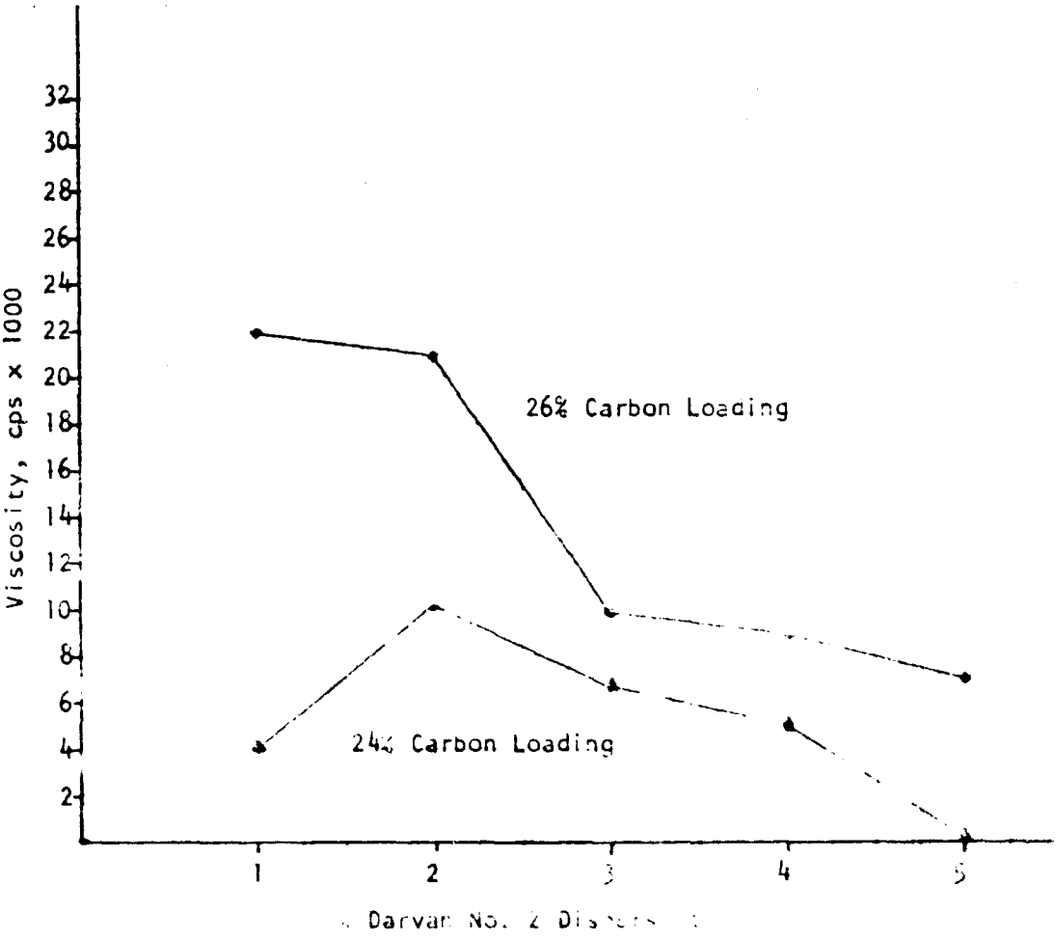

FIGURE 2 - EFFECT OF % DISPERSANT ON DARCO NO GRADE LAMPBLACK

TABLE XII

CARBON SUSPENSIONS
DARCO KB - INHIBITORS

FORMULATION NO.	101	102	108
COMPOSITION BY/WT.			Actual Wt.
Carbon DARCO KB	300 g	300 g	300 g
Thixotrope (1) Bentonite	12 g	12 g	12 g
(2)			
(3)			
(4)			
Dispersant (1) DAXAD 11	12 g	12 g	12 g
(2)			
(3)			
(4)			
Pigment 1100 Yellow	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_2O_4$	6 g		12 g
(2) $Na_2S_2O_3$		6 g	
Solvent, Water/Alcohol 60/40 By Vol.	856 ML	856 ML	856 ML
MIXING TECHNIQUE: Homogenizer, min.	30	30	30
CHARACTERISTICS			
Viscosity, CP			
Imed. After Mix — Helipath "C"	21,000	16,000	16,000
Imed. After Mix — #4 @ 10 RPM	19,200	14,500	14,350
Imed. After Mix — #4 @ 20 RPM	9,600	7,675	7,400
After 24 Hours — Helipath "C"	21,000	16,000	14,000
After 24 Hours — #4 @ 10 RPM	18,400	13,900	13,000
After 24 Hours — #4 @ 20 RPM	9,200	7,450	6,800
After 7 Days — Helipath "C"	19,000	16,000	15,000
After 7 Days — #4 @ 10 RPM	16,200	13,825	13,750
After 7 Days — #4 @ 20 RPM	8,750	7,425	7,150
After 14 Days — Helipath "C"	19,800	16,000	15,000
After 14 Days — #4 @ 10 RPM	17,050	14,200	13,640
After 14 Days — #4 @ 20 RPM	9,200	7,650	7,075
After 30 Days — Helipath "C"	22,000	16,000	14,000
After 30 Days — #4 @ 10 RPM	19,100	15,000	13,200
After 30 Days — #4 @ 20 RPM	11,400	7,950	6,500
pH	8.8	6.1	8.0

NOTES:

TABLE XIII

CARBON SUSPENSIONS
DARCO KB - MIXING TIME EVALUATION

FORMULATION NO.	159	189	199	--	--
COMPOSITION BY/WT.			Actual Wt.		
Carbon Darco KB	325 g	325 g	325 g	325 g	325 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g	6 g
(2)					
(3)					
(4)					
Dispersant (1) Darvan No. 2	36 g	36 g	36 g	36 g	36 g
(2)					
(3)					
(4)					
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	6 g	6 g	6 g	6 g
(2)					
Solvent, Water/Alcohol 60/40 By Vol. ml	856	856	856	856	856
MIXING TECHNIQUE : Homogenizer, Minutes	30	60	90	120	180
CHARACTERISTICS					
Viscosity, CP					
Imed. After Mix — Helipath "C"	14,000	55,000	94,000		
Imed. After Mix — #4 @ 10 RPM	11,900	30,000	45,000		
Imed. After Mix — #4 @ 20 RPM	5,925	22,000	33,000		
After 24 Hours — Helipath "C"	10,000	42,000			
After 24 Hours — #4 @ 10 RPM	8,900	27,000			
After 24 Hours — #4 @ 20 RPM	4,550	19,500			
After 7 Days — Helipath "C"	8,500	42,000			
After 7 Days — #4 @ 10 RPM	7,900	36,000			
After 7 Days — #4 @ 20 RPM	4,250	19,600			
After 14 Days — Helipath "C"	8,000	43,000			
After 14 Days — #4 @ 10 RPM	7,400	37,000			
After 14 Days — #4 @ 20 RPM	3,825	18,500			
After 30 Days — Helipath "C"	8,500	44,000			
After 30 Days — #4 @ 10 RPM	8,000	28,500			
After 30 Days — #4 @ 20 RPM	4,175	20,500			
pH					
NOTES: Solvent Content, %	59.4	58.4	--		

TABLE XIV

CARBON SUSPENSIONS
DARCO KB - PIGMENT EVALUATION

FORMULATION NO.	159	206	207	208
COMPOSITION BY/WT.			Actual Wt.	
Carbon Darco KB	325 g	325 g	325 g	325 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g
(2)				
(3)				
(4)				
Pigment (1) 1100 Yellow	60 g	65 g		
(2) Azo Yellow			65 g	
(3) Cad Yellow/Cad Orange				56/5 g
(4)				
Dispersant: Darvan No. 2	36 g	36 g	36 g	36 g
Corrosive Inhibitor (1) Na_2CrO_4	6 g	6 g	6 g	6 g
(2)				
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756
MIXING TECHNIQUE: Homogenizer, minutes	30	30	30	30
CHARACTERISTICS				
Viscosity, CP				
Imed. After Mix Helipath "C"	14,000	32,000	76,000	15,000
Imed. After Mix #4 @ 10 RPM	11,900	27,000	After	13,500
Imed. After Mix #4 @ 20 RPM	5,925	14,300	2 min.	16,500
After 24 Hours Helipath "C"	10,000	24,000		
After 24 Hours #4 @ 10 RPM	8,900	23,700		
After 24 Hours #4 @ 20 RPM	4,550	11,850		
Color, Wet	Olive Green	Olive Green	Black	Dark Green
Color, Dry	Olive Green	Olive Green	Black	Dark Green
pH				

NOTES:

3. ACC Carbon - Petroleum Coke Based

The petroleum based carbon was supplied by Union Carbide in granular form. The pH value in aqueous solution measured 10.4. The surface area is 980-1080 square meters/gram with a CCL_4 activity of 63%.

As discussed in the Second Quarterly Progress Report, a 25 pound batch of ACC carbon was ball milled for 48 hours. The average particle size after milling was 0.7 μ as determined by the Sub-Sieve Analyzer. Although this particle size is slightly less than desired (1-2 μ), slurries were prepared for testing the thixotropic and dispersing agents. An additional 25 pound batch of ACC was prepared to the desired 1-2 μ concurrently with the experiments and was used to establish final selection of ingredients.

Experiments 98, 99, 122, 123, 124, and 125 as shown in Table XV were prepared to test the efficiency of the bentonite, asbestos, pyrogenic silica and microcrystalline nylon as thixotropes. As the results show, bentonite was the most efficient thixotrope by giving the highest viscosities. The data also shows the effect of particle sizes on both the viscosity and stability during storage. By comparing experiments 99 and 122 it can be seen that slurries prepared with ACC carbon with an average particle size of 1.4 μ remains more stable during storage than those prepared with 0.7 μ carbon particles.

After selecting bentonite as the best thixotrope with ACC carbon, slurries were prepared to evaluate each of the dispersants. Results are shown in Table XVI with carbon loadings of 36.3%. Experiment 127 using Marasperse CB gave the lowest viscosity with ACC petroleum based carbon and was used in further evaluation.

It should be noted that results reported in Table XVI were obtained using ACC carbon with an average particle size of 0.7 μ, all of which show unacceptable stability. This series of experiments to select the most efficient dispersant was re-run using ACC carbon with a particle size of 1-2 μ.

To prepare the desired particle size, a 25 pound batch of ACC carbon was pulverized using the Mikro-Pulverizer and then ball milled for 4-1/2 hours. The average particle size after milling was 1.10 μ as determined by the Sub-Sieve Analyzer.

Using Bentonite as the thixotrope, slurries were prepared to evaluate the various dispersants with the ACC carbons. As shown in Table XVI, Nopcosant K dispersant produces an unmixable slurry, thus was eliminated from further consideration. Experiments 167, 168, and 169, shown in Table XVII, were conducted to investigate the most efficient dispersant. Although Marasperse CB gave the lowest viscosity, Experiment 167, with a carbon loading of 36.3%, attempts to raise the viscosity by additional

carbon were unsuccessful. Carbon loadings over 36.3% result in the formation of heavy glue-like texture that would be impractical to spray. Dispersants DAXAD 11 and Marasperse CB produced slurries that form the gel-like suspension after 24 hour storage.

By comparing Experiments 167 and 169, it can be seen that DAXAD 11 dispersant produces a slurry that increases in viscosity during storage, whereas the Darvan No. 2 slurry will decrease during storage. Since a decrease in viscosity during storage is more desirable for a spray application than is an increase, the Darvan No. 2 was selected as the optimum dispersant.

After selecting the Darvan No. 2 dispersing agent for use with the ACC carbon, Experiments 180, 181, 182, 183, and 186 were conducted to determine the optimum percent. As shown in Table XVIII, slurries containing 2, 3, and 4 percent of Darvan No. 2 gave no significant decreases is viscosity, however in order to produce a thixotrope capable of holding 36% carbon in suspension, it was necessary to increase the thixotrope (bentonite) content from .45% to .67%. These results are also charted in Figure III.

Table XIX show results of evaluating sodium chromate and sodium silicate as corrosion inhibitors in the ACC carbon slurries. Although the sodium chromate resulted in a higher pH, the sodium silicate suspension gave a higher degree of stability during storage. A preliminary selection of siduum chromate was made based on the higher pH, however additional storage data proved sodium silicate to be the superior corrosion inhibitor.

The effect of additional mixing times was next evaluated on the ACC suspensions. As shown in Table XX, additional mixing produced the least changes in viscosity of the four different based carbons evaluated. After three (3) hours the increase was only 6,000 centipoise with approximately 4% of the solvent being lost due to evaporation. Here again the gel-like structure was formed after 24 hours storage with the thirty (30) minute mixing time. Unlike any of the other carbon suspensions tested, mixing time with the ACC suspension can be used to produce a viscosity desirable for spraying.

ACC carbon slurries containing sodium silicate as the corrosion inhibitor show a very high degree of stability. As can be seen by Table XXI, ACC suspensions prepared with sodium silicate do not change in viscosity after static storage for 30 days and longer. Similar suspensions prepared with sodium chromate show a sharp drop in viscosity during a 30 day storage time. Although the pH value is slightly lower in suspensions containing sodium silicate, ($\sim$ 8.0) it is felt that the high degree of stability is the more important property.

TABLE XV

CARBON SUSPENSIONS
ACC CARBON - THIXOTROPES

FORMULATION NO.	98	99	122	123	124	125
COMPOSITION BY/WT.			Actual Wt.			
Carbon ACC	400[1] g	480[2] g	480[3] g	480[3] g	480[3] g	480[3] g
Thixotrope (1) Bentonite	6 g	6 g	6 g			
(2) RG-244				6 g		
(3) COK-84					6 g	
(4) Nylon-6 (Powder)						6 g
Dispersant (1) DAXAD 11	12 g	12 g	12 g	12 g	12 g	12 g
(2) Darvan No. 2						
(3) Marasperse CB						
(4) Nopcosant K						
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) Na_2CrO_4	6 g	6 g	6 g	6 g	6 g	6 g
(2)						
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE : Homogenizer, min.	30	30	30	30	30	30

CHARACTERISTICS

Viscosity, CP							
Imed. After Mix	Helipath "C"	7,000	13,000	13,000	8,000	6,000	4,000
	#4 @ 10 RPM	5,500	11,700	8,500	4,500	4,000	2,200
	#4 @ 20 RPM	2,800	6,100	4,650	2,450	2,150	1,250
After 24 Hours	Helipath "C"	6,000	12,500	25,000	19,000	11,000	9,000
	#4 @ 10 RPM	5,300	11,000	18,000	16,100	6,700	6,700
	#4 @ 20 RPM	2,750	5,825	9,150	7,950	3,450	3,250
After 7 Days	Helipath "C"	6,700	12,500	23,000	19,000	10,000	8,000
	#4 @ 10 RPM	5,300	11,200	17,200	16,000	6,775	5,300
	#4 @ 20 RPM	2,775	5,900	8,700	7,950	3,350	2,700
After 14 Days	Helipath "C"	7,000	13,000	27,000	19,000	10,000	9,000
	#4 @ 10 RPM	5,500	10,500	19,400	14,850	7,100	6,200
	#4 @ 20 RPM	2,850	5,600	10,100	7,600	3,550	3,200
After 30 Days	Helipath "C"	7,000	12,000	34,000	30,000	16,000	13,500
	#4 @ 10 RPM	5,700	10,400	27,200	23,200	9,300	10,450
	#4 @ 20 RPM	2,900	5,650	14,000	12,400	4,625	5,150
pH		11.0	10.8	9.2	9.2	9.3	9.2

NOTES:
1. Ball milled 24 hours - Particle Size Average 1.65 μ in 1-2 lb. batch size
2. Ball milled 48 hours - Particle Size Average 1.40 μ in 1-2 lb. batch size
3. Ball milled 24 hours - Particle Size Average 0.70 μ in 25 lb. batch size

TABLE XVI

CARBON SUSPENSIONS

ACC CARBONS - DISPERSANTS

FORMULATION NO.	122	126	127	127-A	128		
COMPOSITION BY/WT.			Actual Wt.				
Carbon ACC	480 g	480 g	480 g	528 g	480 g		
Thixotrope (1) Bentonite	6 g	6 g	6 g	12 g	6 g		
(2)							
(3)							
(4)							
Dispersant (1) DAXAD 11	12 g						
(2) Darvan No. 2		12 g					
(3) Marasperse CB			12 g	12 g			
(4) Nopcosant K					12 g		
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g		
Corrosive Inhibitor (1) $Na_2C_2O_4$	6 g	6 g	6 g	6 g	6 g		
(2)							
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML		
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30		
CHARACTERISTICS							
Viscosity, CP							
Imed. After Mix — Helipath "C"	13,000	34,000	2,000	38,000			
Imed. After Mix — #4 @ 10 RPM	8,500	28,800	900	19,000			
Imed. After Mix — #4 @ 20 RPM	4,650	15,300	575	10,350			
After 24 Hours — Helipath "C"	25,000	22,000	1,000	17,000			
After 24 Hours — #4 @ 10 RPM	18,000	18,350	500	7,950			
After 24 Hours — #4 @ 20 RPM	9,150	9,600	325	4,700	-		
After 7 Days — Helipath "C"	23,000	14,000	500	7,000	-		
After 7 Days — #4 @ 10 RPM	17,200	10,850	250	3,700	- Discarded - too viscous to mix		
After 7 Days — #4 @ 20 RPM	8,700	5,700	175	2,300			
After 14 Days — Helipath "C"	27,000	12,000	400	6,000			
After 14 Days — #4 @ 10 RPM	19,400	9,950	200	2,900			
After 14 Days — #4 @ 20 RPM	10,100	5,150	175	1,800			
After 30 Days — Helipath "C"	34,000	14,500	500	7,000	-		
After 30 Days — #4 @ 10 RPM	27,200	12,000	300	3,700			
After 30 Days — #4 @ 20 RPM	14,000	6,150	225	2,250			
pH	9.2	9.3	9.2	9.3			

NOTES: FORMULATION MADE WITH .7 % CARBON

TABLE XVII

CARBON SUSPENSIONS
ACC CARBON - DISPERSANTS

FORMULATION NO.	167	168	169
COMPOSITION BY/WT.			Actual Wt.
Carbon ACC	480 g	480 g	480 g
Thixotrope (1) Bentonite	6 g	6 g	6 g
(2)			
(3)			
(4)			
Dispersant (1) Darvan No. 2	12 g		
(2) Marasperse CB		12 g	
(3) DAXAD 11			12 g
(4) Nopcosant K	- Eliminated - (Ref. Formulation 128)		
Pigment 1100 Yellow	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	6 g	6 g
(2)			
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756
MIXING TECHNIQUE : Homogenizer, minutes	30	30	30

CHARACTERISTICS

Viscosity, CP				
Imed. After Mix	Helipath "C"	13,000	1,500	10,000
	#4 @ 10 RPM	8,000	700	7,400
	#4 @ 20 RPM	4,350	475	4,000
After 24 Hours	Helipath "C"	9,000	1,000	12,000
	#4 @ 10 RPM	5,600	500	10,450
	#4 @ 20 RPM	3,100	350	5,450
After 7 Days	Helipath "C"	4,900	400	13,500
	#4 @ 10 RPM	2,950	300	10,300
	#4 @ 20 RPM	1,750	225	5,350
After 14 Days	Helipath "C"	4,000	300	15,000
	#4 @ 10 RPM	2,450	250	11,500
	#4 @ 20 RPM	1,500	200	5,900
After 30 Days	Helipath "C"	6,000		15,000
	#4 @ 10 RPM	2,850	Settled	14,600
	#4 @ 20 RPM	1,700		7,400
pH		9.1	9.3	9.2

NOTES: Formulations prepared with 1.10 carbon.

TABLE XVIII

CARBON SUSPENSIONS
ACC CARBON - PERCENT DISPERSANT

FORMULATION NO.		180	181	182	183		186	
COMPOSITION BY/WT.				Actual Wt.				
Carbon ACC		480 g	480 g	480 g	480 g		480 g	
Thixotrope (1)	Bentonite	6 g	6 g	6 g	6 g		9 g	
(2)								
(3)								
(4)								
Dispersant (1)	Darvan No. 2	12 g	24 g	36 g	48 g		24 g	
(2)								
(3)								
(4)								
Pigment	1100 Yellow	60 g	60 g	60 g	60 g		60 g	
Corrosive Inhibitor (1)	$Na_2C_rO_4$	6 g	6 g	6 g	6 g		6 g	
(2)								
Solvent, Water/Alcohol 60/40 By Vol., ml		756	756	756	756		756	
MIXING TECHNIQUE: Homogenizer, minutes		30	30	30	30		30	
CHARACTERISTICS								
Viscosity, CP								
Immed. After Mix	Helipath "C"	14,000	1,800	1,500	2,000		9,000	
	#4 @ 10 RPM	8,150	900	900	1,225		5,250	
	#4 @ 20 RPM	4,650	650	625	825		3,225	
After 24 Hours	Helipath "C"	7,200	700	800	1,000		4,000	
	#4 @ 10 RPM	4,400	500	500	625		2,300	
	#4 @ 20 RPM	2,650	375	375	475		1,500	
After 7 Days	Helipath "C"							
	#4 @ 10 RPM							
	#4 @ 20 RPM							
After 14 Days	Helipath "C"							
	#4 @ 10 RPM							
	#4 @ 20 RPM							
After 30 Days	Helipath "C"							
	#4 @ 10 RPM							
	#4 @ 20 RPM							
pH								

NOTES: Formulations prepared with 1.10 ± carbon.

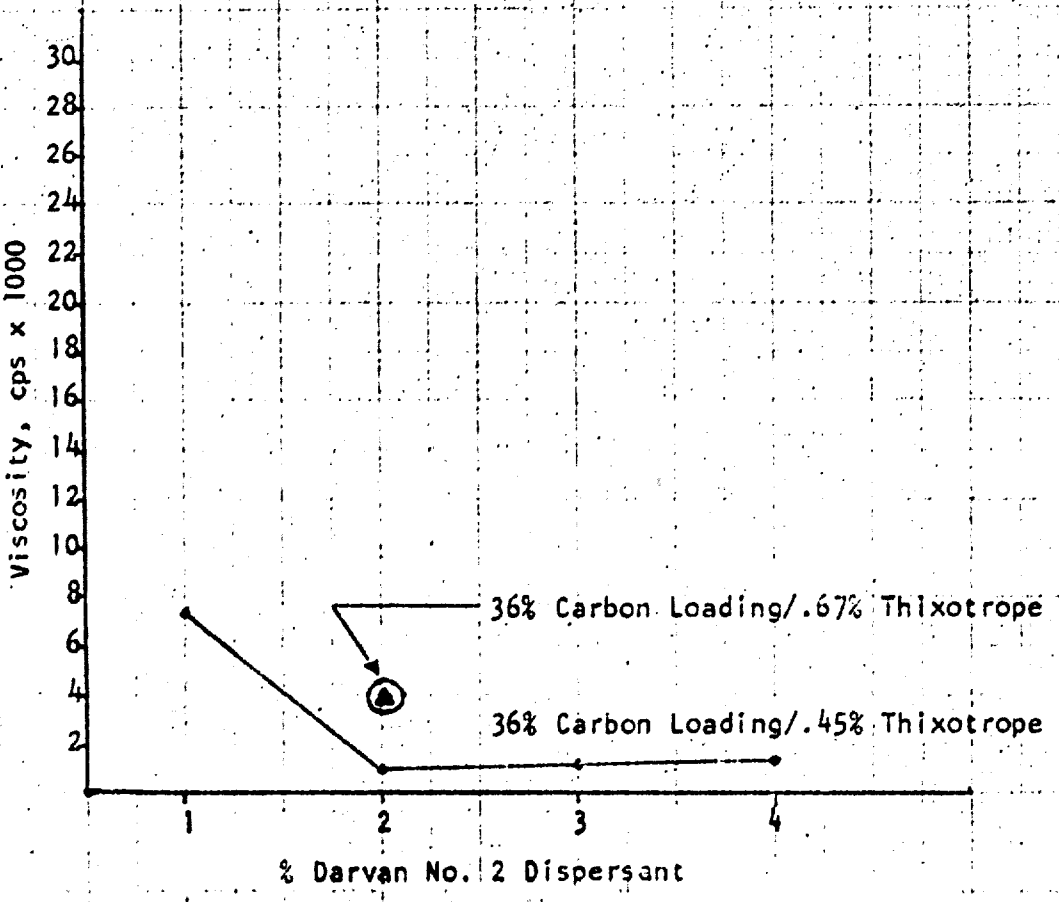

FIGURE 3 - EFFECT OF % DISPERSANT ON ACC CARBON SLURRIES

TABLE XIX

CARBON SUSPENSIONS
ACC CARBON - CORROSION INHIBITOR

FORMULATION NO.	186	187
COMPOSITION BY/WT.		Actual Wt.
Carbon ACC	480 g	480 g
Thixotrope (1) Bentonite	9 g	9 g
(2)		
(3)		
(4)		
Dispersant (1) Darvan No. ?	24 g	24 g
(2)		
(3)		
(4)		
Pigment 1100 Yellow	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	
(2) $Na_2S_iO_3$		6 g
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756
MIXING TECHNIQUE : Homogenizer, minutes	30	30

CHARACTERISTICS

Viscosity, CP			
Init. After Mix	Helipath "C"	9,000	6,000
	#4 @ 10 RPM	5,250	3,900
	#4 @ 20 RPM	3,225	2,400
After 24 Hours	Helipath "C"	4,000	5,500
	#4 @ 10 RPM	2,300	3,650
	#4 @ 20 RPM	1,500	2,300
After 7 Days	Helipath "C"	1,500	6,000
	#4 @ 10 RPM	1,100	4,500
	#4 @ 20 RPM	850	2,750
After 14 Days	Helipath "C"	1,000	6,000
	#4 @ 10 RPM	850	4,400
	#4 @ 20 RPM	700	2,725
After 30 Days	Helipath "C"	1,000	6,000
	#4 @ 10 RPM	800	4,500
	#4 @ 20 RPM	700	2,800
pH		8.9	8.0

NOTES: Particle Size 1.10 μ

TABLE XX

CARBON SUSPENSIONS
ACC CARBON - MIXING TIME EVALUATION

FORMULATION NO.		186	190	198	200	202
COMPOSITION BY/WT.				Actual Wt.		
Carbon ACC		480 g	480 g	480 g	480 g	480 g
Thixotrope (1) Bentonite		9 g	9 g	9 g	9 g	9 g
(2)						
(3)						
(4)						
Dispersant (1) Darvan No. 2		24 g	24 g	24 g	24 g	24 g
(2)						
(3)						
(4)						
Pigment 1100 Yellow		60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$		6 g	6 g	6 g	6 g	6 g
(2)						
Solvent, Water/Alcohol 60/40 By Vol., ml		756	756	756	756	756
MIXING TECHNIQUE: Homogenizer, minutes		30	60	90	120	180
CHARACTERISTICS						
Viscosity, CP						
Imed. After Mix	Helipath "C"	9,000	8,000	10,500	11,000	16,000
	#4 @ 10 RPM	5,250	5,250	6,600	7,100	8,800
	#4 @ 20 RPM	3,225	3,200	4,000	4,350	5,300
After 24 hours	Helipath "C"	4,000	4,500	7,000	6,000	10,000
	#4 @ 10 RPM	2,300	2,700	4,200	3,400	6,100
	#4 @ 20 RPM	1,500	1,800	2,700	2,300	3,900
After 7 Days	Helipath "C"	1,500	2,000	3,800	5,000	7,000
	#4 @ 10 RPM	1,100	1,500	2,350	3,350	4,150
	#4 @ 20 RPM	850	1,100	1,700	2,400	2,775
After 14 Days	Helipath "C"	1,000	1,500	2,500	4,000	5,000
	#4 @ 10 RPM	850	1,100	1,900	2,800	3,500
	#4 @ 20 RPM	700	900	1,450	2,075	2,550
After 30 Days	Helipath "C"	1,000	1,500	3,000	6,000	6,000
	#4 @ 10 RPM	800	1,300	2,100	3,700	5,900
	#4 @ 20 RPM	700	1,050	1,625	2,700	2,800
pH		8.9	9.0	9.0	9.0	9.0
Solvent Content, %		55.7				57.6
Particle Size: 1.10						

TABLE XXI

CARBON SUSPENSIONS
ACC CARBON - CORROSION INHIBITORS

FORMULATION NO.	186	190		187	211	217
COMPOSITION BY/WT.				Actual Wt.		
Carbon ACC	480 g	480 g		470 g	470 g	380 g
Thixotrope (1) Bentonite	9 g	9 g		9 g	9 g	9 g
(2)						
(3)						
(4)						
Dispersant (1) Darvan No. 2	24 g	24 g		24 g	24 g	24 g
(2)						
(3)						
(4)						
Pigment 1100 Yellow	60 g	60 g		68 g	68 g	68 g
Corrosive Inhibitor (1) Na_2CrO_4	6 g	6 g				
(2) $Na_2S_iO_3$				6 g	6 g	6 g
Solvent, Water/Alcohol 60/40 By Vol.	756	756		756	756	756
MIXING TECHNIQUE : Homogenizer, Minutes	30	60		30	30	
CHARACTERISTICS						
Viscosity, CP						
Imed. After Mix — Helipath "C"	9,000	8,000		6,000	6,000	2,500
#4 @ 10 RPM	5,250	5,250		3,900	4,000	1,425
#4 @ 20 RPM	3,225	3,200		2,400	2,350	850
After 24 hours — Helipath "C"	4,000	4,500		6,000	6,000	2,500
#4 @ 10 RPM	2,300	2,700		4,000	3,900	1,400
#4 @ 20 RPM	1,500	1,800		2,300	2,300	850
After 7 Days — Helipath "C"	1,500	2,000		6,000	6,000	1,900
#4 @ 10 RPM	1,100	1,500		4,500	3,850	1,000
#4 @ 20 RPM	850	1,100		2,750	2,300	600
After 14 Days — Helipath "C"	1,000	1,500		6,000	5,500	1,900
#4 @ 10 RPM	850	1,100		4,400	3,700	1,000
#4 @ 20 RPM	700	900		2,725	2,300	650
After 30 Days — Helipath "C"	1,000	1,500		6,000		
#4 @ 10 RPM	800	1,300		4,500		
#4 @ 20 RPM	700	1,050		2,800		
pH	9.9	9.0		8.0	8.0	6.0

NOTES: Particle Size: 1.10 μ

Based on the results discussed above, using the ACC carbon, the following selection can be made.

Ingredient	Actual Weight	Percent Formula Wt.
Carbon: ACC	470 g	34.99
Thixotrope: Bentonite	9 g	.67
Dispersant: Darvan No. 2	24 g	1.79
Pigment: 1100 Yellow	78 g	5.81
Corrosion Inhibitor: Sodium Silicate	6 g	.45
Solvent: (60/40 Water-Methanol)	756 ml	56.29
Mixing Time: Thirty (30) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours after mixing	6,000 cps Helipath 4,000 cps #4 @ 10 RPM 2,300 cps #4 @ 20 RPM	
pH Value	8.0	
Carbon Particle Size	1.1 μ (avg.)	

4. UU Carbon - Coconut Shell Based

The coconut shell based carbon used for this evaluation was supplied by Barnebey-Cheney Company in 50 x 200 mesh size particles. The pH value measured 10.4 in aqueous solution. The surface area is 14-16,000 square meters/gram with a CCL_4 activity of 100%.

In order to perform the required number of experiments, a 50 lb. batch of UU was ball milled for 16 hours. The average particle size as measured by the Sub-Sieve Analyzer was 0.7 μ. This carbon was used to prepare suspension for testing bentonite, asbestos, pyrogenic silica and nylon thixotropic agents. As the data in Table XXII shows, bentonite gave the highest viscosities and is considered to be the most efficient. In considering particle size and their effect on viscosities, experiments 100 and 129 show that it is possible to attain a much higher carbon loading with 1.2 μ than 0.7 μ. Also, the viscosity of the slurry containing the 1.2 μ is lower and more stable than the one containing the 0.7 μ carbon. Additional UU carbon was milled to the desired 1-2 μ particle size and used in making final selection of materials.

With bentonite being the most efficient thixotrope with the UU carbon, suspensions were prepared to evaluate each of the dispersants. Experiments 129, 138, 139, and 140 shown in Table XXIII give the results of this evaluation. Using a carbon loading of 32.2% (paricle size 0.7 μ) in each experiment, Marasperse CB appeared to give a better dispersion of carbon particles indicated by the low viscosity. Considering the effect of particle size on percent carbon loading, viscosity and stability, this series of experiments performed to select the most efficient dispersant was repeated using UU carbon with a particle size in the 1-2 μ range.

A 25 pound batch of UU carbon was pulverized using the Mikro-Pulverizer and then ball milled for 4-1/2 hours. The average particle size as measured by the Sub-Sieve Analyzer was 1.50 μ. Having selected bentonite as the most efficient thixotrope, suspensions were prepared to evaluate the dispersants. Here again as Table XXIII shows, the Nopcosant K dispersant gave unsatisfactory dispersions and was eliminated from further study. Experiment 161, 164, 165, 166, 170 and 171, listed in Table XXIV show Darvan No. 2 to be the most efficient dispersant. Using a carbon loading of 31% (particle size 1.50 μ) Darvan No. 2 appeared to give the best dispersion with the lowest viscosity.

Experiments 166, 170 and 171 using a concentration of 2% Darvan No. 2, show that a maximum carbon loading of 34.8% is possible for this particular dispersant.

After selecting Darvan No. 2 as the dispersing agent with UU carbon, the optimum percentage of this dispersant was next evaluated and is shown in Table XXV. As this series of experiments show, additional amounts of dispersant over 2% had very little effect on viscosities. These results are also shown in Figure IV.

Experiments shown in Table XXVI were conducted to evaluate sodium chromate and sodium silicate as corrosion inhibitors. By comparing Experiments 170 and 179 it can be seen that suspensions prepared with sodium silicate had much higher viscosities than did the slurries containing sodium chromate with only slight increase in pH value. Sodium chromate was selected as the corrosion inhibitor.

The effect of mixing time was next evaluated using a 2% concentration of Darvan No. 2 dispersant. Results listed in Table XXVII show that increases in mixing time give corresponding increase in viscosity. It appears that a mix time of sixty (60) minutes will be sufficient to provide a uniform slurry that will form a gel-like structure after 24 hours and will hold the 34.8% carbon in suspension.

Table XXVIII show results of evaluating various pigments to produce an olive green. Concentrations up to ∿ 5% of the cadmium lithopones gave a gradient from dark green to black. Approximately ∿ 5% (4.95%) of the Mapico 1100 yellow gave the desired olive green.

Although suspensions containing 34.68% UU carbon have been prepared and are sprayable, stabilization of viscosity during storage remains a problem. Unlike the CWS, where the viscosity tends to raise during storage, the UU suspensions show a sharp decrease during the first 24 hours after mix and continues to drop during storage. Although the thixotropic gel will hold the carbon in suspension and is stable from that standpoint, attempts to stabilize the viscosity over 30 days and longer storage were unsuccessful.

TABLE XXII

CARBON SUSPENSIONS

UU CARBON - THIXOTROPES

FORMULATION NO.	100	129	130	131	132
COMPOSITION BY/WT.			Actual Wt.		
Carbon UU	400[1] g	350[2] g	350[2] g	350[2] g	350[2] g
Thixotrope (1) Bentonite	6 g	6 g			
(2) RG-244			6 g		
(3) COK-84				6 g	
(4) Nylon-6					6 g
Dispersant (1) DAXAD 11	12 g	12 g	12 g	12 g	12 g
(2) Darvan No. 2					
(3) Marasperse CB					
(4) Nopcosant K					
Pigment 1100 Yellow	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_1O_4$	6 g	6 g	6 g	6 g	6 g
(2) $Na_2S_1O_3$					
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML	756 ML
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30	30
CHARACTERISTICS					
Viscosity, CP					
Imed. After Mix — Helipatn "C"	17,000	18,000	15,200	13,000	11,000
Imed. After Mix — #4 @ 10 RPM	13,500	16,200	14,800	12,900	10,800
Imed. After Mix — #4 @ 20 RPM	6,900	8,400	7,900	6,450	5,525
After 24 Hours — Helipath "C"	15,000	21,500	13,000	16,000	13,700
After 24 Hours — #4 @ 10 RPM	10,800	19,300	17,800	15,500	13,000
After 24 Hours — #4 @ 20 RPM	5,650	9,350	9,400	7,800	6,800
After 7 Days — Helipath "C"	18,000	28,000	18,800	21,000	14,000
After 7 Days — #4 @ 10 RPM	14,400	22,400	17,550	18,300	13,800
After 7 Days — #4 @ 20 RPM	7,400	11,700	9,350	9,450	7,225
After 14 Days — Helipath "C"	20,000	33,000	18,000	21,000	13,000
After 14 Days — #4 @ 10 RPM	15,000	25,400	16,900	16,000	12,900
After 14 Days — #4 @ 20 RPM	7,775	13,250	8,950	8,600	6,750
After 30 Days — Helipath "C"	22,000	41,000	17,000	23,000	13,000
After 30 Days — #4 @ 10 RPM	18,400	28,400	16,500	14,100	12,700
After 30 Days — #4 @ 20 RPM	9,500	15,200	8,900	9,000	6,550
pH	10.2	9.4	9.6	9.6	9.6

NOTES: 1. Ball mill 64 hours - Particle Size Average 1.2 - 1-2 lb batch size

2. Ball mill 16 hours - Particle Size Average [illegible] - 50 lb batch size

TABLE XXIII

CARBON SUSPENSIONS
UU CARBON - DISPERSANTS

FORMULATION NO.	129	138	139	140			
COMPOSITION BY/WT.			Actual Wt.				
Carbon UU	350 g	350 g	350 g	350 g			
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g			
(2)							
(3)							
(4)							
Dispersant (1) DAXAD 11	12 g						
(2) Marasperse CB		12 g					
(3) Darvan No. 2			12 g				
(4) Nopcosant K				12 g			
Pigment 1100 Yellow	60 g	60 g	60 g	60 g			
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	6 g	6 g	6 g			
(2)							
Solvent, Water/Alcohol 60/40 By Vol.	756 ML	756 ML	756 ML	756 ML			
MIXING TECHNIQUE: Homogenizer, min.	30	30	30	30			
CHARACTERISTICS							
Viscosity, CP							
Imed. After Mix — Helipath "C"	18,000	4,000	8,700	35,000			
Imed. After Mix — #4 @ 10 RPM	16,200	2,800	7,300	29,000			
Imed. After Mix — #4 @ 20 RPM	8,400	1,500	3,900	14,900			
After 24 Hours — Helipath "C"	21,500	3,500	6,000	29,000			
After 24 Hours — #4 @ 10 RPM	19,300	1,800	4,700	19,700			
After 24 Hours — #4 @ 20 RPM	9,350	1,000	2,425	12,900			
After 7 Days — Helipath "C"	28,000	2,500	4,000	21,500			
After 7 Days — #4 @ 10 RPM	22,400	1,500	3,000	18,000			
After 7 Days — #4 @ 20 RPM	11,700	850	1,600	10,600			
After 14 Days — Helipath "C"	33,000	3,400	4,000	19,000			
After 14 Days — #4 @ 10 RPM	25,400	1,600	2,700	16,500			
After 14 Days — #4 @ 20 RPM	13,250	950	1,475	9,500			
After 30 Days — Helipath "C"	41,000	5,000	4,900	17,000			
After 30 Days — #4 @ 10 RPM	26,400	2,000	2,700	16,400			
After 30 Days — #4 @ 20 RPM	15,200	1,150	1,500	8,950			
pH	9.4	9.4	9.5	9.8			

NOTES: PARTICLE SIZE 0.7 μ

TABLE XXIV

CARBON SUSPENSIONS
UU CARBON - DISPERSANT EVALUATION

FORMULATION NO.	161	164	165		166	170	171
COMPOSITION BY/WT.			Actual Wt.				
Carbon UU	385 g	385 g	385 g		400 g	455 g	455 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	- eliminated - (Ref Formulation 140)	6 g	6 g	6 g
(2)							
(3)							
(4)							
Dispersant (1) DAXAD 11	12 g						
(2) Darvan No. 2		12 g			24 g	24 g	
(3) Marasperse CB			12 g				24 g
(4) Nopcosant K				- E -			
Pigment 1100 Yellow	60 g	60 g	60 g		60 g	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	6 g	6 g		6 g	6 g	6 g
(2)							
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756		756	756	756
MIXING TECHNIQUE: Homogenizer, minutes	30	30	30		30	30	30
CHARACTERISTICS							
Viscosity, CP							
Imed. After Mix — Helipath "C"	12,000	8,500	9,000		6,000	13,000	19,000
Imed. After Mix — #4 @ 10 RPM	9,400	5,600	6,600		3,825	9,400	14,300
Imed. After Mix — #4 @ 20 RPM	4,800	2,950	3,400		2,075	5,150	7,400
After 24 Hours — Helipath "C"	14,000	4,000	7,000		2,300	3,500	5,000
After 24 Hours — #4 @ 10 RPM	9,900	2,600	4,500		1,450	1,900	2,600
After 24 Hours — #4 @ 20 RPM	5,175	1,475	2,400		850	1,275	1,675
After 7 Days — Helipath "C"	26,000	3,700	5,000		400	1,200	2,000
After 7 Days — #4 @ 10 RPM	16,200	2,100	2,200		300	850	1,300
After 7 Days — #4 @ 20 RPM	8,550	1,200	1,300		225	625	900
After 14 Days — Helipath "C"	31,000	5,000	4,500		- Settled -	800	1,000
After 14 Days — #4 @ 10 RPM	19,000	2,800	2,000			550	700
After 14 Days — #4 @ 20 RPM	10,000	1,600	1,150			475	600
After 30 Days — Helipath "C"	44,000	9,000	3,500			500	600
After 30 Days — #4 @ 10 RPM	22,000	4,600	1,800			450	500
After 30 Days — #4 @ 20 RPM	13,600	2,500	1,175			425	400
pH	9.6	9.6	9.6		9.5	9.5	9.7

NOTES: Particle Size: 1.50 μ

TABLE XXV

CARBON SUSPENSIONS
UU CARBON - PERCENT DISPERSANT EVALUATION

FORMULATION NO.	173	170	174	175
COMPOSITION BY/WT.			Actual Wt.	
Carbon UU	455 g	455 g	455 g	455 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6
(2)				
(3)				
(4)				
Dispersant (1) Darvan No. 2	12 g	24 g	36 g	48 g
(2)				
(3)				
(4)				
Pigment 1100 Yellow	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1) Na_2CrO_4	6 g	6 g	6 g	6 g
(2)				
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756
MIXING TECHNIQUE : Homogenizer, minutes	30	30	30	30
CHARACTERISTICS				
Viscosity, CP				
Imed. After Mix — Helipath "C"	28,000	13,000	13,000	14,000
Imed. After Mix — #4 @ 10 RPM	20,000	9,400	8,600	9,250
Imed. After Mix — #4 @ 20 RPM	13,000	5,150	4,750	5,100
After 24 Hours — Helipath "C"	16,000	3,500	4,000	3,900
After 24 Hours — #4 @ 10 RPM	13,200	1,900	2,300	2,300
After 24 Hours — #4 @ 20 RPM	6,800	1,275	1,450	1,425
After 7 Days — Helipath "C"				
After 7 Days — #4 @ 10 RPM				
After 7 Days — #4 @ 20 RPM				
After 14 Days — Helipath "C"				
After 14 Days — #4 @ 10 RPM				
After 14 Days — #4 @ 20 RPM				
After 30 Days — Helipath "C"				
After 30 Days — #4 @ 10 RPM				
After 30 Days — #4 @ 20 RPM				
pH				

NOTES:

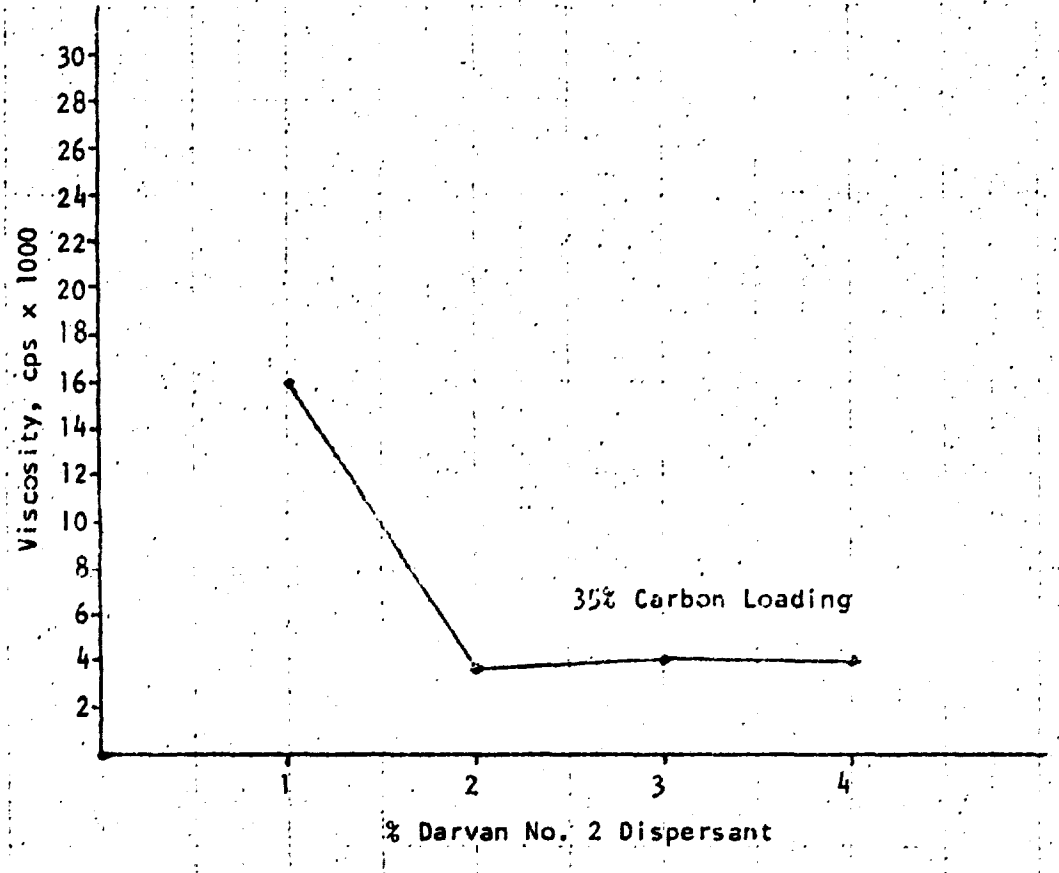

FIGURE 4 - EFFECT OF % DISPERSANT ON UU CARBON SLURRIES

TABLE XXVI

CARBON SUSPENSIONS
UU CARBON - CORROSION INHIBITOR

FORMULATION NO.	170	179
COMPOSITION BY/WT.		Actual Wt.
Carbon UU	455 g	455 g
Thixotrope (1) Bentonite	6 g	6 g
(2)		
(3)		
(4)		
Dispersant (1) Darvan No. 2	24 g	24 g
(2)		
(3)		
(4)		
Pigment 1100 Yellow	60 g	60 g
Corrosive Inhibitor (1) $Na_2C_rO_4$	6 g	
(2) $Na_2S_iO_3$		6 g
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756
MIXING TECHNIQUE : Homogenizer, minutes	30	30

CHARACTERISTICS

	Viscosity, CP		
Imed. After Mix	Helipath "C"	13,000	62,000
	#4 @ 10 RPM	9,400	21,800
	#4 @ 20 RPM	5,150	13,250
After 24 hours	Helipath "C"	3,500	54,000
	#4 @ 10 RPM	1,900	22,200
	#4 @ 20 RPM	1,275	12,000
After 7 Days	Helipath "C"	1,200	
	#4 @ 10 RPM	850	
	#4 @ 20 RPM	625	
After 14 Days	Helipath "C"	800	- Thick - Discarded -
	#4 @ 10 RPM	550	
	#4 @ 20 RPM	475	
After 30 Days	Helipath "C"	500	
	#4 @ 10 RPM	450	
	#4 @ 20 RPM	425	
pH		9.5	9.7

NOTES:

TABLE XXVII

CARBON SUSPENSIONS
UU CARBON - MIXING TIME EVALUATION

FORMULATION NO.		170	194	195	196	197
COMPOSITION BY/WT.				Actual Wt.		
Carbon	UU	455 g	455 g	455 g	455 g	455 g
Thixotrope (1)	Bentonite	6 g	6 g	6 g	6 g	6 g
(2)						
(3)						
(4)						
Dispersant (1)	Darvan No. 2	24 g	24 g	24 g	24 g	24 g
(2)						
(3)						
(4)						
Pigment	1100 Yellow	60 g	60 g	60 g	60 g	60 g
Corrosive Inhibitor (1)	$Na_2C_rO_4$	6 g	6 g	6 g	6 g	6 g
(2)						
Solvent, Water/Alcohol 60/40 By Vol., ml		756	756	756	756	756
MIXING TECHNIQUE: Homogenizer, minutes		30	60	90	120	180
CHARACTERISTICS						
Viscosity, CP						
Imed. After Mix	Helipath "C"	13,000	27,000	21,500	30,000	49,000
	#4 @ 10 RPM	9,400	18,800	16,900	22,000	29,000
	#4 @ 20 RPM	5,150	10,050	9,000	11,800	16,000
After 24 Hours	Helipath "C"	3,500	9,000	10,000	15,500	24,000
	#4 @ 10 RPM	1,900	6,900	7,600	11,000	19,800
	#4 @ 20 RPM	1,275	3,975	4,300	6,200	10,800
After 7 Days	Helipath "C"	1,200	5,000	7,000	13,000	20,500
	#4 @ 10 RPM	850	3,500	4,700	8,000	15,100
	#4 @ 20 RPM	625	2,250	2,950	4,725	8,400
After 14 Days	Helipath "C"	800	4,000	6,000	10,000	23,000
	#4 @ 10 RPM	550	3,000	4,500	7,600	17,400
	#4 @ 20 RPM	475	1,950	2,850	4,600	9,700
After 30 Days	Helipath "C"	500	5,000	10,000	16,000	34,000
	#4 @ 10 RPM	450	3,550	6,300	11,500	22,700
	#4 @ 20 RPM	425	2,350	3,900	6,750	12,400
pH		9.5	9.6	9.6	9.6	9.6
NOTES: Solvent Content, %		58.9				57.9

TABLE XXVIII

CARBON SUSPENSIONS
UU CARBON - PIGMENT EVALUATION

FORMULATION NO.	170	203	204	205
COMPOSITION BY/WT.			Actual Wt.	
Carbon UU	455 g	455 g	455 g	455 g
Thixotrope (1) Bentonite	6 g	6 g	6 g	6 g
(2)				
(3)				
(4)				
Pigment (1) 1100 Yellow	60 g	65 g		
(2) Cad Yellow/Cad Orange			56/5 g	
(3) Azo Yellow				65 g
(4)				
Dispersant: Darvan No. 2	24 g	24 g	24 g	24 g
Corrosive Inhibitor (1) Na_2CrO_4	6 g	6 g	6 g	6 g
(2)				
Solvent, Water/Alcohol 60/40 By Vol., ml	756	756	756	756
MIXING TECHNIQUE: Homogenizer, Minutes	30	30	30	30

CHARACTERISTICS

Viscosity, CP					
Imed. After Mix.	Helipath "C"	13,000	33,000	29,000	44,000
	#4 @ 10 RPM	8,600	22,000	19,800	17,400
	#4 @ 20 RPM	4,750	11,800	10,400	10,600
After 24 Hours	Helipath "C"	4,000	10,000	8,500	38,000
	#4 @ 10 RPM	2,300	7,900	6,200	15,400
	#4 @ 20 RPM	1,450	4,550	3,625	7,800
Color, Wet		Olive Green	Olive Green	Dark Green	Black
Color, Dry		Olive Green	Olive Green	Dark Green	Black
pH					

NOTES:

The optimum formulation for the UU coconut shell based carbon is made as follows:

Ingredient	Actual Weight	Percent Formula Wt.
Carbon: UU	455 g	34.68
Thixotrope: Bentonite	6 g	.46
Dispersant: Darvan No. 2	24 g	1.83
Pigment: 1100 Yellow	65 g	4.95
Corrosion Inhibitor: Sodium Chromate	6 g	.46
Mix Time: Sixty (60) minutes using the L-1 Laboratory Homogenizer		
Typical Viscosity at 24 hours after mixing	8,000 cps Helipath	
	6,000 cps #4 @ 10 RPM	
	3,450 cps #4 @ 20 RPM	
pH Value	1.5 μ (avg.)	

D. The Formation of Thixotropic Gels

In working with the four different based carbons, it has been observed that during the first three days storage of certain carbon slurries, a thixotropic, non-pourable, uniform gel is formed. This is considered a very important and desirable condition in that it prevents the settling out of the carbon particles. It is believed that this gel forms as a result of the interaction of bentonite with the active carbon.

This gel-like structure is very shear sensitive and can be easily made fluid by one or two shakes of the container.

It can be concluded from the data presented in Table XXIX that slurries with high undisturbed viscosities as high as 102,000 centipoise (and possibly higher) can be easily reconstituted into a fluid state with lowered viscosities that can be easily sprayed. For example, Formulation No. 138 with an undisturbed viscosity of 102,000 centipoise reconstitutes easily to 6,000 centipoise.

Of equal importance is the fact that the gel-like structure will reform repeatedly after being reconstituted to the fluid state.

TABLE XXIX

FORMATION OF A THIXOTROPIC GEL

Form # CWS	Viscosity Undist.	Viscosity Reconstitution[1]	Amount Free Solvent	Amount Settling	Gel Formation L-M-H[2]	Color	Easily Reconstituted	Notes
115	70,000	16,000	20 ml	0	Yes (L)	Green	Yes	
116	--	--	100 ml	100%	No (H)	--	No	
117	136,000	8,000	82 ml	50%	Yes (M)	Green	Yes	
118	--	--	--	100%	No (H)	Green	No	
120	40,000	1,200	23 ml	0	Yes (L)	Green	Yes	
137	>200,000	3,000	6 ml	0	Yes (H)	Green	No	Very heavy glue-like
141	140,000	2,000	3 ml	0	Yes (M)	Green	Yes	
144	74,000	2,000	8 ml	0	Yes (L)	Green	Yes	
145	163,000	34,000	11 ml	0	Yes (H)	Green	Yes	Hard gel bu easily re-constitutec
ACC[3]								
122	65,000	30,000	6 ml	0	Yes (L)	Black	Yes	
123	58,000	24,000	15 ml	0	Yes (I)	Black	Yes	
124	74,000	14,000	20 ml	0	Yes (L)	Black	Yes	
125	50,000	12,000	27 ml	0	Yes (L)	Black	Yes	
126	70,000	17,000	9 ml	0	Yes (M)	Black	Yes	

(1) Re-constituted. Helipath

(2) Light, Medium, Hard

(3) Suspension Prepared with .7 u Particle Size Carbon

TABLE XXIX (CONT'D)

FORMATION OF A THIXOTROPIC GEL

Form # UU	Viscosity Undist.	Viscosity Reconstitution(1)	Amount Free Solvent	Amount Settling	Gel Formation L-M-H(2)	Color	Easily Reconstituted	Notes
100	>200,000	30,000	9 ml	0	Yes (M)	Green	Yes	
129	136,000	50,000	6 ml	0	Yes (M)	Green	Yes	
130	110,000	15,000	34 ml	0	Yes (M)	Green	Yes	
131	>200,000	27,000	34 ml	0	Yes (M)	Green	Yes'	
132	100,000	10,000	39 ml	0	Yes (L)	Green	Yes	
138	102,000	6,000	21 ml	0	Yes (L)	Green	Yes	
139	74,000	5,000	16 ml	0	Yes (L)	Green	Yes	
140	82,000	17,500	18 ml	0	Yes (L)	Green	Yes	
DARCO KB								
97	57,000	19,000	7 ml	0	Yes (L)	Green	Yes	
104	>200,000	11,000	62 ml	30%	No (H)	Green	Yes	
106	>200,000	15,000	44 ml	30%	No (H)	Green	No	
112	44,000	19,000	7 ml	0	Yes (L)	Green	Yes	
113	30,000	9,500	5 ml	0	Yes (L)	Green	Yes	
105	29,000	5,000	13 ml	0	Yes (L)	Green	Yes	
133	90,000	36,000	2 ml	0	Yes (L)	Green	Yes	
136	104,000	28,000	2 ml	0	Yes (L)	Green	Yes	
142	130,000	32,000	2 ml	0	Yes (L)	Green	Yes	
143	108,000	28,000	6 ml	0	Yes (L)	Green	Yes	

III. SPRAYING AND ADHESION TESTING

A. Spray Experiments

To begin spraying experiments with each of the optimized formulations, two 24" x 24" aluminum test panels were spray coated with 1 mil of chromate primer and 1 mil of OG lusterless alkyd lacquer. Test tabs (1 in^2) were coated along with the panel and were used for determining the amount of carbon deposited on the surface after each test. The standard sprayer unit equipped with the standard nozzle and .033" diameter atomizing hole was used to test each of the different based carbons. Figure V and VI show the spray set-up and coated test panel.

Spray Test No. 12 - CWS Carbon

The optimized formulation as discussed in Section I-C was used for this test. The viscosity measured in the 14,000 centipoise range. The standard sprayer was filled with 1 liter and pressurized to 200 psi from a nitrogen (standard 2500 cubic feet) tank. The 24" x 24" test panel was set 90° to the sprayer nozzle at a distance of 5 feet. One pass was made over the entire panel. As the slurry tends to be broken up into very small droplets, one pass was not sufficient to cover the surface of the panel. Two additional passes were then made which covered the surface completely. After allowing the coating to dry, the test tabs were removed to determine coverage.

Test Tab No.	Carbon Deposited
1	22.4 mg/cm^2
2	19.2 mg/cm^2
3	21.9 mg/cm^2
4	19.6 mg/cm^2

Because the slurry is being dispensed in very small droplets, requiring 2-3 passes for complete surface coverage, high carbon deposits are being recorded as shown above.

Spray Test No. 13 - Darco KB Carbon

The same general procedure was used to test sprayability of the Darco KB suspensions. The viscosity measured in the 17,000 centipoise range. A total of 3 passes was made over the panel with the following coverage.

Test Tab No.	Carbon Deposited
1	12.6 mg/cm^2
2	11.8 mg/cm^2
3	10.4 mg/cm^2
4	9.7 mg/cm^2

Again the spray was in the form of very small droplets requiring multiple passes for complete surface coverage.

Spray Test No. 14 - ACC Carbon

Although the ACC suspension sprayed better than did the other carbon slurries, the surface coating was similar to that of the previous tests. Again the spray was in the form of small droplets. The viscosity measured in the 6,000 cps range. After 3 passes over the test panel the coverage measured as follows:

Test Tab No.	Carbon Deposited
1	10.6 mg/cm^2
2	10.1 mg/cm^2
3	10.4 mg/cm^2
4	10.8 mg/cm^2

The spray tests conducted on the UU suspension gave results very similar to those obtained using the CWS and Darco KB slurries. Each test tab showed an average of 12 mg/cm^2 carbon coating.

By considering stability and sprayability of the different carbon based thixotropes, the suspension prepared with the ACC activated carbon was chosen for additional spray tests.

Spray Test No. 15 - ACC Carbon

To further test the sprayability of the ACC suspensions, a standard DeVilbiss paint spray gun was used to prepare a test panel. The spray gun was a type MBC with AV15EX head and DEX needle and was operated at 68 psi air pressure.

From a distance of 15-18 inches, 2 passes were made over the test panel. The resulting spray, fog rather than droplets, gave a uniform even coating. The test tabs were weighed for coverage and the following results were obtained.

Test Tab No.	Carbon Deposited
1	4.56 mg/cm^2
2	3.72 mg/cm^2
3	4.43 mg/cm^2
4	3.72 mg/cm^2

For comparison, a spray test was made with the same ACC suspension but spraying with the standard apparatus. From a distance of 5 feet, operating at 200 psi nitrogen pressure, 3 passes were necessary over the test panel to cover the surface. The test tabs measured the following coverage.

Test Tab No.	Carbon Deposited
1	8.2 mg/cm^2
2	7.8 mg/cm^2
3	9.2 mg/cm^2
4	8.1 mg/cm^2

In viewing Figures VII and VIII, it can be seen that a more uniform, even coating is applied using the DeVilbiss gun than was possible with the existing standard spray unit. The panel prepared with the standard apparatus has the larger carbon deposit (8-9 mg/cm^2) but has evidence of uncoated areas caused by the droplet form spray.

It can be concluded from the above tests that with some modification to the standard spray apparatus, the ACC carbon slurries can be sprayed satisfactorily to give the desired 3-4 mg/cm^2 activated carbon coating.

B. Adhesion Tests

Adhesion tests were next performed using formulations as described in Section 1-C. In preparing for abrasion tests, three aluminum panels 3" x 6" were coated with 1 mil of chromate primer and 1 mil of lusterless OG alkyd lacquer. These test panels along with test tabs were then coated with the activated carbon slurry as follows:

Spray Gun:	DeVilbiss Type MBC AV15-EX Nozzle/DEX Needle
Distance:	15-18 inches panel to spray gun
Pressure:	100 pounds air pressure
Passes:	2 in 7-10 seconds

After drying for twelve hours at 72°F and 50% R.H., the test tabs were reweighed to determine coverage. Figures IX and X show the test panel before and after spraying.

Terry cloth strips 3.16 x 3.16 centimeters (10 cm^2 surface area) were cut for abrasion pads. A 10 mil thick rigid plastic film was placed over the terry cloth to evenly distribute the 100 gram weight. The weighed terry cloth was dragged over the carbon coating at a rate of 5 inches per minute using the Instron Test Machine (see Figures XI and XII. The percent carbon loss was calculated as follows:

$$\frac{W_2 - W_1}{P_A} = W_A \ (gm/cm^2)$$

$$\frac{W_2 - W_3}{T_A} = W_B \ (g/cm^2)$$

$$\frac{W_B}{W_A} \times 100 = \% \ (g/cm^2) \text{ Carbon Loss}$$

Where:

W_1 = Weight of test panel

W_2 = Weight of carbon coated test panel

W_A - Amount of carbon coating per unit area

P_A = Area of test panel in cm^2

W_3 = Weight of test panel after abrasion

T_A - Area of the abrasion path in cm^2

W_B - Amount of carbon abraded away

Table XXX shows results of the abrasion tests for each of the different based carbons. It can be seen that all carbons are well within the maximum allowable loss of 25%.

TABLE XXX

ABRASION RESISTANCE OF ACTIVATED CARBON
DEPOSITED ON ALKYD LACQUER COATED SURFACES

Carbon	Test Panel Number	Carbon Coating mg/cm^2	Carbon Loss Percent
1. CWS	1	3.87	3.30
Coal Based	2	3.36	6.06
	3	3.55	2.84
2. Darco KB	10	5.12	4.78
Wood Based	11	5.49	4.58
	12	4.99	5.18
3. ACC	7	4.01	6.36
Petroleum Based	8	4.80	7.19
	9	3.83	6.71
4. UU	4	3.87	12.09
Coconut Shell Based	5	3.75	8.92
	6	3.27	6.74

IV. CONCLUSIONS

Ingredients for the formulation of an activated carbon loaded thixotrope have been selected. Each of the different based carbons has been evaluated for stability, sprayability, color and adhesion to a surface. Optimized formulations of each system are shown in Section II-C.

Spray tests were conducted with each of the different carbon suspensions for sprayability, coverage and effective distance. The ACC thixotrope was selected as the system best fulfilling the program performance requirements using sprayability, viscosity and stability as the selection criteria.

Adhesion tests were conducted to determine percent retention of carbon on a painted surface after being subject to drag test. It was found that each of the different based carbons has sufficient adherence to remain below the allowable 25% loss.

V. FUTURE WORK

Work during the next reporting period will be as follows:

1. Optimization of spraying apparatus and techniques.

2. Work will begin on dry formulations for clothing and individual equipment decontaminants. These dry forms will contain an activated carbon (CWS), a filler such as asbestos or micronized nylon, and a pigment such as iron oxide. This formulation will be incorporated into a loosely woven pad.

VI. APPENDIX

FIGURE 5 - SPRAY TESTING APPARATUS TO DETERMINE SPRAYABILITY AND COVERAGE

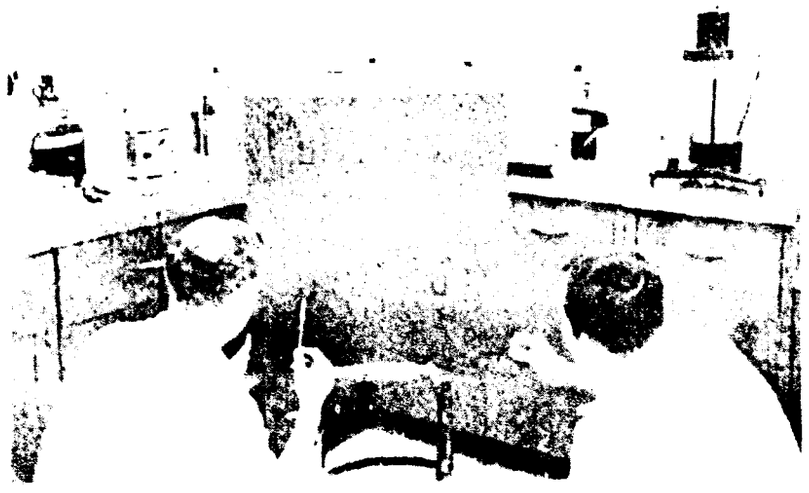

FIGURE 6 - CARBON COATED PANEL WITH TEST TABS TO MEASURE COVERAGE

FIGURE 7 - CLOSE-UP OF ACTIVATED CARBON COATING SPRAYED WITH THE STANDARD SPRAYER APPARATUS

FIGURE 8 - CLOSE-UP OF ACTIVATED CARBON COATING SPRAYED WITH A DEVILBISS MBC SPRAY GUN

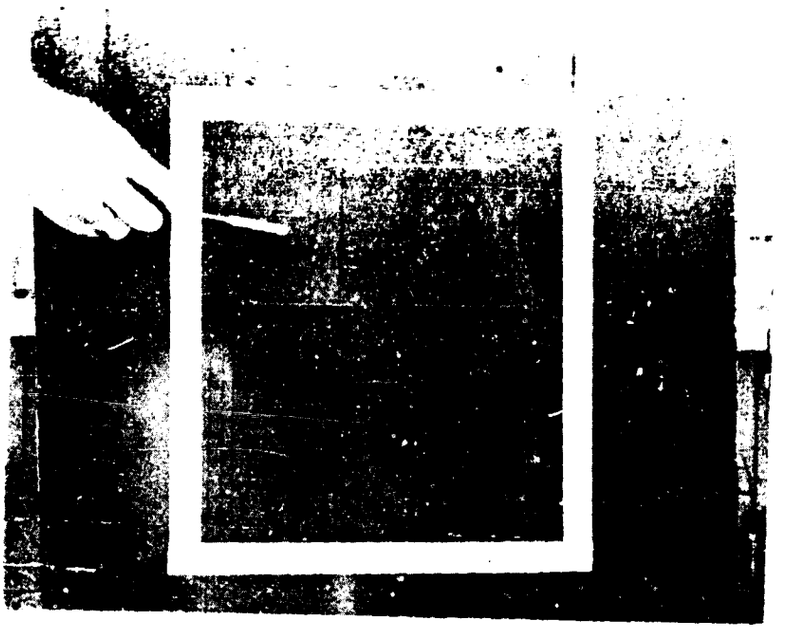

FIGURE 6 - CARBON COATED PANEL WITH TEST TABS
TO MEASURE COVERAGE

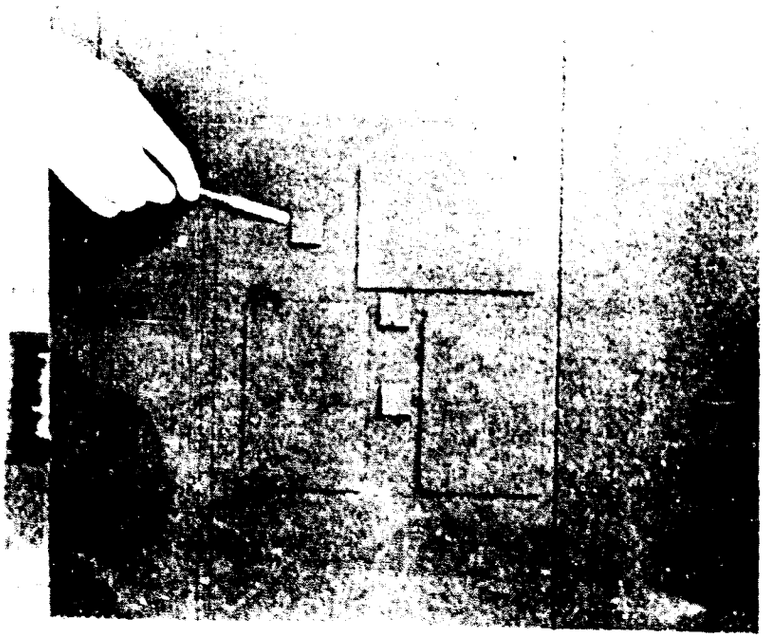

FIGURE 10 - ABRASION TE T PANELS WITH A 3-4 mg/cm^2
COATING OF AC IVATED CARBON

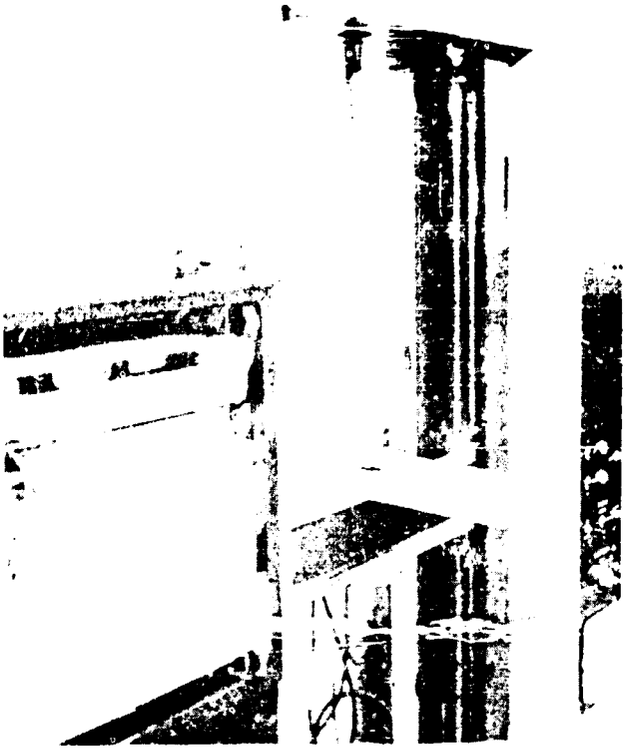

FIGURE 11 - ABRASION TEST SET-UP
USING INSTRON TEST MACHINE

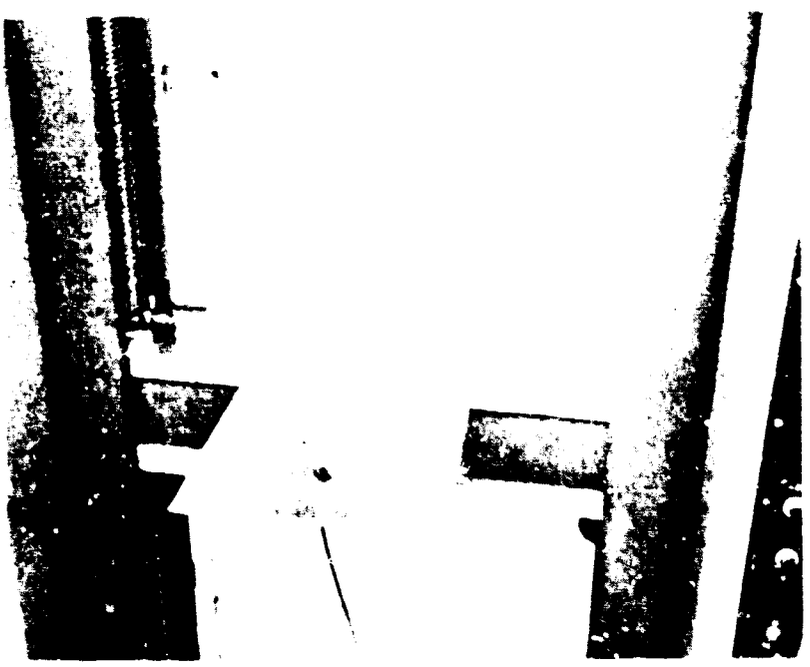

FIGURE 12 - CLOSE-UP SHOWING 100 GRAM
WEIGHT AND TERRY CLOTH