Unclassified
Security Classification

DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY *(Corporate author)* Union Carbide Corporation Tarrytown, New York	2a. REPORT SECURITY CLASSIFICATION Unclassified 2b. GROUP

3. REPORT TITLE

DEVELOPMENT OF AN IMPROVED DUST-CONTROL SYSTEM BASED ON POLYVINYL ACETATE LATEX

4. DESCRIPTIVE NOTES *(Type of report and inclusive dates)*

Final report

5. AUTHOR(S) *(First name, middle initial, last name)*

D. F. Anderson
J. A. Durante
L. H. Wartman

6. REPORT DATE October 1971	7a. TOTAL NO. OF PAGES 53	7b. NO. OF REFS None

8a. CONTRACT OR GRANT NO. DACA 39-70-C-0011 b. PROJECT NO. c. d.	9a. ORIGINATOR'S REPORT NUMBER(S) 9b. OTHER REPORT NO(S) *(Any other numbers that may be assigned this report)* U. S. Army Engineer Waterways Experiment Station Contract Report S-71-9

10. DISTRIBUTION STATEMENT

Approved for public release; distribution unlimited.

11. SUPPLEMENTARY NOTES Prepared under contract for U. S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi	12. SPONSORING MILITARY ACTIVITY U. S. Army Materiel Command Washington, D. C.

13. ABSTRACT

While dusting and the formation of dust clouds have always been significant factors in military operations, the advent of modern large-scale airborne tactics has transformed what was once primarily an annoyance to a costly and serious problem because dust ingestion into moving parts of aircraft accelerates wear, resulting in increased maintenance time and reduced operational availability. The U. S. Army Engineer Waterways Experiment Station (WES) has undertaken the task of finding methods for suppressing dust. One of the systems developed employs DCA-70, a commercial dust-control agent manufactured by Union Carbide Corporation. The DCA-70 is sprayed on a graded soil surface where it forms a continuous film, anchored to the substrate with good resistance to damage by air blasts and vehicular traffic. DCA-70 is a polyvinyl acetate latex compounded with dibutyl phthalate at a concentration of 10% of the contained solids in the latex. The dibutyl phthalate acts as a plasticizer, making films cast from the latex more pliable and resistant to rupture. Films cast from DCA-70 are very resistant to deterioration by the actinic radiation in sunlight and hence, the outdoor-life expectancy of stabilized soils would be expected to be good. However, because the dibutyl phthalate is quite volatile, it evaporates in hot climates resulting in decreased resiliency and strength. Since many commercial plasticizers of much lower volatility than dibutyl phthalate are available, a program was undertaken to find a plasticizer system which could be substituted for the dibutyl phthalate to yield a product with better aging characteristics. In Phase I of this project more than thirteen plasticizers were tested for compatibility with the polyvinyl acetate latex used in the manufacture of DCA-70. Those plasticizers which showed the most promise in the initial screening evaluation were selected for further trials involving preparation of laboratory latex samples, casting of films, and measurement of tensile properties both before and after oven aging and water extraction tests. As expected, all the products made with low volatility plasticizers retained their tensile properties much better than DCA-70. On the basis of the test results six formulations were selected for preparation of 110-gallon quantities and field evaluation by WES. Their evaluation has resulted in the elimination of four of these formulations from consideration. A selection remains to be made between the two remaining candidates, both of which are made with a mixed system containing FLEXOL 4-GO and Santicizer 140, but in different concentrations. WES evaluation confirms that the modified versions are superior to DCA-70. In Phase II of this project study, nine specification test methods, most of which are standard ASTM procedures for latex products, were applied to various commercial polyvinyl acetate latexes after addition of plasticizers using the formulations developed in Phase I of the contract research. Comparison of the data obtained with those attributes expected to be of importance for good performance in dust-control applications leads to a set of tentative specification values which should allow procurement of these products on a competitive basis. The tests used are tensile properties of films, total solids, surface tension, pH, viscosity, filterable solids, salt tolerance, storage stability at 140 F, and shear stability. In addition, it is recommended that only vinyl acetate homopolymers plasticized by the recommended formula be employed in this application.

DD FORM 1 NOV 65 **1473** REPLACES DD FORM 1473, 1 JAN 64, WHICH IS OBSOLETE FOR ARMY USE.

Unclassified
Security Classification

Unclassified

Security Classification

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Dust control						
Dustproofing						
Plasticizers						
Polyvinyl acetate latex						
Soil stabilization						

Unclassified

Security Classification

CONTRACT REPORT S-71-9

DEVELOPMENT OF AN IMPROVED DUST-CONTROL SYSTEM BASED ON POLYVINYL ACETATE LATEX

by

D. F. Anderson, J. A. Durante, L. H. Wartman

October 1971

Sponsored by U. S. Army Materiel Command

Conducted for U. S. Army Engineer Waterways Experiment Station, Vicksburg, Mississippi

Under Contract No. DACA 39-70-C-0011

By Union Carbide Corporation, Tarrytown, New York

ARMY MRC VICKSBURG, MISS.

APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED

FOREWORD

The studies described in this report were performed under contract number DACA 39-70-C-0011 for research and development work on polyvinyl acetate dust-control systems. The contract, dated 19 December 1969, is between the U. S. Army Engineer Waterways Experiment Station (WES), Vicksburg, Mississippi, and Union Carbide Corporation. There were two phases to the contract work. Phase I is entitled "Development of an Improved Dust-Control System Based on Polyvinyl Acetate Latex", and Phase II is called "Development of Procurement Specifications for an Improved Dust-Control System Based on Polyvinyl Acetate Latex". The Summary section describes both phases of the work. However, for reasons of clarity and convenience, the body of the report treats each phase separately.

The studies were conducted in the Tarrytown Technical Center Laboratories of the Union Carbide Corporation, Tarrytown, New York, under the supervision of Mr. L. H. Wartman, Group Leader, with consulting advice from Mr. W. M. Sullivan, Development Scientist. Phase I was carried out by Dr. D. F. Anderson, while Phase II was done by Mr. J. A. Durante.

The contract was monitored by Mr. Royce C. Eaves, Chief, Stabilization Section, Expedient Services Branch, under the general supervision of Mr. J. P. Sale, Chief, Soils Division, WES. Contracting Officer was COL E. D. Peixotto, CE.

TABLE OF CONTENTS

TABLE OF CONTENTS (CON'T.)

TABLE OF CONTENTS (CONT.)

Tables

SUMMARY

While dusting and the formation of dust clouds have always been significant factors in military operations, the advent of modern large-scale airborne tactics has transformed what was once primarily an annoyance to a costly and serious problem because dust ingestion into moving parts of aircraft accelerates wear, resulting in increased maintenance time and reduced operational availability. The U. S. Army Engineer Waterways Experiment Station (WES) has undertaken the task of finding methods for suppressing dust. One of the systems developed employs DCA-70, a commercial dust-control agent manufactured by Union Carbide Corporation. The DCA-70 is sprayed on a graded soil surface where it forms a continuous film, anchored to the substrate, with good resistance to damage by air blasts and vehicular traffic.

DCA-70 is a polyvinyl acetate latex compounded with dibutyl phthalate at a concentration of 10% of the contained solids in the latex. The dibutyl phthalate acts as a plasticizer, making films cast from the latex more pliable and resistant to rupture. Films cast from DCA-70 are very resistant to deterioration by the actinic radiation in sunlight and hence, the outdoor-life expectancy of stabilized soils would be expected to be good. However, because the dibutyl phthalate is quite volatile, it evaporates in hot climates resulting in decreased resiliency and strength. Since many commercial plasticizers of much lower volatility than dibutyl phthalate are available, a program was undertaken to find a plasticizer system which could be substituted for the dibutyl phthalate to yield a product with better aging characteristics.

In Phase I of this project more than thirteen plasticizers were tested for compatibility with the polyvinyl acetate latex used in the manufacture of DCA-70. Those plasticizers which showed the most promise in the initial screening evaluation were selected for further trials involving preparation of laboratory latex samples, casting of films, and measurement of tensile properties both before and after oven aging and water extraction tests. As expected, all the products made with low volatility plasticizers retained their tensile properties much better than DCA-70. On the basis of the test

results six formulations were selected for preparation of 110-gallon quantities and field evaluation by WES. Their evaluation has resulted in the elimination of four of these formulations from consideration. A selection remains to be made between the two remaining candidates, both of which are made with a mixed system containing FLEXOL 4-GO and Santicizer 140, but in different concentrations. WES evaluation confirms that the modified versions are superior to DCA-70.

In Phase II of this project study, nine specification test methods, most of which are standard ASTM procedures for latex products, were applied to various commercial polyvinyl acetate latexes after addition of plasticizers using the formulations developed in Phase I of the contract research. Comparison of the data obtained with those attributes expected to be of importance for good performance in dust-control applications leads to a set of tentative specification values which should allow procurement of these products on a competitive basis.

The tests used are tensile properties of films, total solids, surface tension, pH, viscosity, filterable solids, salt tolerance, storage stability at 140° F., and shear stability. In addition, it is recommended that only vinyl acetate homopolymers plasticized by the recommended formula be employed in this application.

DEVELOPMENT OF AN IMPROVED DUST-CONTROL SYSTEM BASED ON POLYVINYL ACETATE LATEX (PHASE I)

INTRODUCTION

DCA-70 has been used successfully as a dust-control material in experimental tests by WES. In their test procedure, a mixture of two parts DCA-70 with one part water was sprayed on test plots of various soil types. A tough protective film formed with sufficient soil penetration to anchor the film to the soil and prevent the formation of dust clouds. Subjecting such protected areas to simulated and actual aircraft landings and light vehicular traffic has shown good results. Although DCA-70 was found to be superior to many other products evaluated, extended exposure to hot, dry climates resulted in an embrittled film. This was attributed to the loss of plasticizer. Since DCA-70 contains a relatively volatile plasticizer, dibutyl phthalate, it seemed that better aging stability could be achieved by substituting a less volatile product, many of which are available commercially.

The test work on finding a replacement for dibutyl phthalate was carried out by obtaining from Union Carbide manufacturing facilities samples of the latex ordinarily used in making DCA-70. This latex is a homopolymer of vinyl acetate with a total solids content of about 58% by weight, a specific gravity at 20°C of 1.104, and a maximum residual vinyl acetate content of 0.6% by weight. Various technical difficulties prevent accurate determination of the molecular weight of the polymer but viscosity measurements on the polymer dissolved in dimethylformamide indicate it is relatively high in molecular weight. The size of the polymer particles in the latex is relatively large. Typical size distributions as measured by a Coulter counter show 6% of the particles with a diameter of under 0.75 microns. Seventy per cent range in diameter from 0.75 to 2.75 microns, and the remainder range in particle size up to 4.25 microns. The latex also contains various ingredients such as surfactants and protective colloids dissolved in the aqueous phase.

EXPERIMENTAL PROCEDURES

A. Plasticizer Screening for Compatibility - A series of thirteen plasticizers

(see Table 1) was screened to seek possible replacement candidates for the volatile dibutyl phthalate currently used in DCA-70. The screening procedure consisted in thoroughly mixing 100 g. of the precursor polyvinyl acetate emulsion (60% solids) with 6 g. of the plasticizer (10 phr; 10 parts plasticizer per 100 parts resin). The polyvinyl acetate emulsion - plasticizer mixture was stirred for five minutes with a mechanical blade stirrer. Films of 0.005" to 0.007" thickness were cast on glass plates by pouring the emulsion on the leveled plate, spreading to uniform thickness with a doctor blade, and allowing to dry. After drying some of the films could be readily removed from the glass plates while others were brittle compared to a control specimen of DCA-70. The following numerical values were assigned to each plasticizer sample with the control sample containing dibutyl phthalate (FLEXOL DBP) assigned an arbitrary efficiency value of 1°.

1° - Good strong elastic film
2° - Moderately good film, slightly brittle
3° - Poor film, very brittle

Those samples which were assigned a value of 3° were immediately dropped from the program. The remaining samples were subjected to further testing.

Further evaluation of the plasticizers with efficiency values of 2° included experiments in which the basic formulation was varied by incorporating 5 phr of butyl CARBITOL acetate (BCA) as a coalescing agent. Mixing, film casting, and evaluations were performed as described above for the initial screening.

B. Physical Test Procedures - Several physical test procedures were designed to evaluate the experimental plasticized formulations against the dibutyl phthalate plasticized emulsion (DCA-70) as the control. These tests were: (1) 140°F oven stability, (2) minimum filming temperature, (3) tensile strength and elongation, and (4) water extractability. Each of these test procedures is described below:

1. Preparation of Samples - The following procedure was followed in preparing the samples and conditioning the films for much of the physical testing. A 100-g. sample of the base latex (60% solids as polyvinyl acetate) was weighed into a 250-ml. pyrex beaker. The appropriate amount of plasticizer was then added, after which the mixture was stirred for 5 minutes at medium speed with a mechanical

stirrer. The sample was then placed in a centrifuge for deaeration. After removal from the centrifuge, four 0.020" films were cast on glass plates. The films were removed from the glass plates after drying (2 hours) and hung in the laboratory at ambient conditions for seven days. Dogbone samples (Type B specimens as described in ASTM D412-64T) were then cut from the films and divided into three groups. Group I was tested for tensile strength and percent elongation. The samples in Group II were placed in a forced-draft oven for various periods to study plasticizer volatility. Group III samples were used to evaluate water extractability. Data are recorded in Tables 2-5.

2. Plasticizer Volatility - Dogbone samples from Group II were weighed to the nearest 0.0001 g. and hung in a forced-draft oven at 140° F for various periods of time. After the desired heating cycle, the samples were removed, cooled to room temperature, and reweighed. The weight loss was assumed to be due to volatilization of the plasticizer. Data are given in Tables 2-5.

3. Water Extractability - Group III films were placed in a water bath at ambient temperatures for various periods of time. After removal, the films were allowed to dry in the laboratory for 24 hours before measuring tensile strength and elongation. Data obtained in this manner are recorded in Tables 2 and 3.

Some water-extractability data were obtained by a modified method in which the samples were exposed to a continuous flow of tap water. In this way saturation of the water by sparingly soluble plasticizers was avoided and test data were more closely related to practical situations such as continuous rain fall. Data obtained in this manner are recorded in Table 4.

4. Tensile Strength and Elongation - Dogbone film specimens from Groups I, II, and III were subjected to tensile and elongation tests as specified in ASTM D638-64T. The tests were run in a constant humidity (50% RH) , constant temperature (23°C) room but difficulties in controlling the humidity were encountered. Polyvinyl acetate picks up water and relative humidity has a considerable effect on tensile test measurements. As an expedient, results on single sets of specimens were obtained on a single day so that all data would be obtained under comparable conditions. As a consequence, in interpreting the data in the tables

(2 - 5), it is recommended that comparisons be made only within a given table. Comparing data obtained on separate days and recorded in separate tables may lead to erroneous conclusions.

5. Minimum Filming Temperature - A portion of the latex prepared in procedure 1 was used for minimum filming-temperature measurements. A 0.010" film of latex was spread on an aluminum block heated at one end and cooled at the other so as to yield a temperature gradient over the length of the block from -10°C to 50°C. After 30 minutes drying, the latex forms a continuous film only at temperatures sufficiently high to allow coalescence of the individual emulsified polymer particles. The position on the block corresponding to the minimum temperature at which a continuous film was formed was noted and the temperature at this position was measured with a contact pyrometer. Table 3 records the results.

6. Filterable Solids - Some of the experimental samples were tested for filterable solids in order to ascertain whether any large particles formed on addition of various plasticizers. The samples (1000 g.) were diluted with three times their volume of cold tap water and stirred to ensure homogeneity. They were then filtered through a 60-mesh screen which had been weighed to provide a tare weight. Fresh water was poured through the screen until all foam and milkiness disappeared. The screens were then dried at 135°C for one hour, cooled, and weighed. The filterable solids content was calculated by the equation:

$$\frac{\text{Final weight} - \text{Tare weight}}{1000 \times \%\ \text{solids in latex}} \times 10^4 = \text{Percent filterable solids by weight}$$

Filterable solids data are recorded in Table 6.

7. Viscosity - Viscosity data were obtained using a Brookfield viscometer. Details of the measurement techniques employed are given in the report on Phase II of this document. See Section F under Experimental Procedures.

TEST RESULTS

A. Plasticizer Screening for Compatibility - Several plasticizers were selected and evaluated to determine their compatibility with and their ability to plasticize the polyvinyl acetate emulsion. These materials (shown in Table 1) were selected for their relatively low vapor pressure and high boiling point characteristics as compared to dibutyl phthalate. This evaluation was conducted in order to find possible replacement candidates for dibutyl phthalate in DCA-70. Most of the experimental replacement candidates were found to be incompatible with the polyvinyl acetate emulsion. When formulations incorporating these plasticizers were prepared, the resulting films were extremely brittle and could not be removed from glass plates. FLEXOL TCP, FLEXOL A-26, FLEXOL Z-88, Santicizer 140, and Benzoflex 9-88 provided marginal film-forming properties and were selected for further evaluation. FLEXOL 3-GH and FLEXOL 4-GO were found to be compatible with the latex precursor and equal to or better than DBP in film-forming characteristics. The films formed on the glass plates were very flexible and easily removed without cracking.

The plasticizer candidates listed as marginal (2°) were used in a formulation incorporating butyl CARBITOL acetate (BCA) as a coalescing agent since the inefficiency of plasticization could conceivably be due to failure to imbibe the plasticizer into the emulsified polymer particles. The results of film forming tests revealed FLEXOL TCP, Santicizer 140, and Benzoflex 9-88 to be potential candidates provided BCA was incorporated to enhance coalescence and plasticizer imbibition.

B. Physical Testing of Plasticized Latex Films - The plasticizers rated 1° and 2° in the cursory study were subjected to further physical testing for the evaluation of tensile properties after aging at ambient conditions, after thermal exposure at 140°F and after water extraction. The results of this study (Table 2) indicated FLEXOL 4-GO (12 phr) and Santicizer 140 (20 phr) to be likely candidates for replacement of dibutyl phthalate, since the tensile properties and elongation before thermal aging or water extraction appeared approximately equal to their DBP-plasticized counterpart. One particularly important observation was the relatively low plasticizer loss during thermal aging in formulations utilizing these two materials: FLEXOL 4-GO at 12 phr 6.3% loss, and Santicizer 140 at 20 phr 3.6% loss. These materials also retained their flexibility (good percent elongation) and tensile strength after thermal aging or water extraction. Films made from DCA-70 under identical conditions, on the other hand, exhibited a 30% loss of the contained DBP plasticizer and became quite brittle.

Those formulations in which BCA was used showed good initial elongation and tensile strength (Table 2) after conditioning at ambient conditions for seven days. However, after the thermal exposure for 100 hours at 140°F, much of the BCA was lost and embrittlement ensued. Further work on these marginal cases was discontinued due to unsatisfactory performance after thermal exposure.

A continued evaluation of the performance of FLEXOL 4-GO and Santicizer 140 in individual formulations and also in formulations utilizing mixtures of the two clearly indicated superior performance over the DCA-70 control (Table 3). These formulations were FLEXOL 4-GO (10 phr), Santicizer 140 (20 phr), and a FLEXOL 4-GO/Santicizer 140 mixture (4.5 phr/10.5 phr). Other mixtures were also investigated but were shown to be less efficient than the above mentioned samples. These candidates showed improved elongation and tensile strength in addition to low plasticizer volatility and negligible water extraction. Minimum filming-temperature data (Table 3) showed these formulations gave a 2°C to 7°C lower coalescing temperature than the DCA-70 control.

In order to retain the plasticizer level at 10 phr with improved physical properties over DCA-70, several FLEXOL 4-GO/Santicizer 140 mixtures with the latex precursor were prepared and evaluated. A formulation incorporating a mixture of Santicizer 140 (3.5 phr) and FLEXOL 4-GO (6.5 phr) was found to be superior to DCA-70 and the other experimental samples. Data on these mixed plasticizer systems are given in Table 4.

Table 4 shows data obtained when the volatility experiments were expanded to 170 and 340 hours and the water-extraction tests to 340 hours. These results present further evidence relating to the superior performance of the experimental samples over the control. The plasticizer loss of the experimental films was relatively low even after 340 hours, compared to the losses for the FLEXOL DBP-plasticized films. Resiliency was retained after 340 hours; whereas, the control films of DCA-70 embrittled after 170 hours. All samples, including DCA-70, performed adequately in the water-extractability evaluation. It is interesting to note that the tensile strengths of these films (see Table 4) were significantly lower than those observed in previous test runs on similar formulations. This was due to excessively high humidity conditions during the testing periods, as discussed earlier.

C. Mixture Characteristics and Aging Study - Further studies on the mixture characteristics and aging during storage of the latex product for several formulations were performed in our South Charleston Laboratories where facilities for preparations of larger quantities than can be made at Tarrytown are available. Table 6 lists the results of these tests. The aging study was conducted at room temperature (25°C.) and at 50°C using the recommended amounts of plasticizers and twice the amount to exaggerate the effect. Sample analyses were made after aging one day at 25°C and again after aging two weeks at 25°C and 50°C. The results show only a slight increase in viscosity and filterable solids of the latex using FLEXOL 4-GO. Essentially 100 percent of the respective plasticizers are retained in the polymer after a total solids measurement (1 hour at 135°C). Thus, the addition of FLEXOL 4-GO and the FLEXOL 4-GO/Santicizer 140 mixtures at experimental target levels appears to be feasible and no difficulties with agglomeration or other aging effects in the stored latex are anticipated.

D. Effect of Antioxidant Additive on Physical Properties of Latex Films - FLEXOL 4-GO (tetraethyleneglycol di-2-ethylhexanoate) contains ether linkages that are subject to oxidative attack which could result in deterioration on extended exposure to sunlight. Although an oxidative degradation problem is not anticipated, the possible use of an antioxidant (Topanol CA at 1.5 phr) was investigated, and the effects on filming properties were determined. No detrimental effects on the physical properties of the film were observed. Visual observations revealed complete incorporation of the antioxidant into the plasticized latex. The resulting tensile strength and elongation after water extraction, oven aging at 140°F, and room temperature aging appeared excellent. Therefore, if conditions warrant the addition of an antioxidant to the formulations, no difficulty is anticipated provided any problems which may be encountered with incorporation of the antioxidant can be solved.

E. Effect of Thickening Agents on Plasticized Latex Formulations - A series of experiments was run in which thickening agents were incorporated into the experimental formulations. Calidria Asbestos RG-244 and DuPont's Ludox HS-40 were tried as thickeners in an attempt to modify flow characteristics of the diluted latex by decreasing the flow at low strain levels. Conceivably, building a thixotropic flow characteristic of this type into the latex could yield applied films of more uniform thickness since there would be less tendency for the latex to thin out at high spots on the graded soil surface. Both materials are effective, but possible field application would be difficult due to handling problems. Also separation of the additive from the latex occurs after standing for 14 hours. Studies utilizing this approach were discontinued.

F. Shear Stability of Latex Formulations - Each of the experimental formulations were subjected to the moderately high shearing force provided by a high-speed food blender. Each sample showed a slight increase in viscosity, but the homogeneous consistency was retained and no agglomerates formed.

G. Viscosity Versus Dilution Curves - A plotted curve of viscosity in centipoise versus percent solids in the latex is recorded in Figure 1. Data are given for only one of the experimental products since all materials gave identical curves. It has been noted in field trials that dispenser loading using pump suction is much easier when the viscosity is reduced by decreasing the solids level to the 55-57% region.

H. Pilot-Plant Production - The results on compatibility and aging performance were used to select six formulations for preparation in 110-gallon quantities for evaluation at the WES in actual soil applications. All formulations are based on FLEXOL 4-GO, Santicizer 140, or combinations of these two materials. Table 5 lists these materials and also summarizes the oven and ambient-aging results.

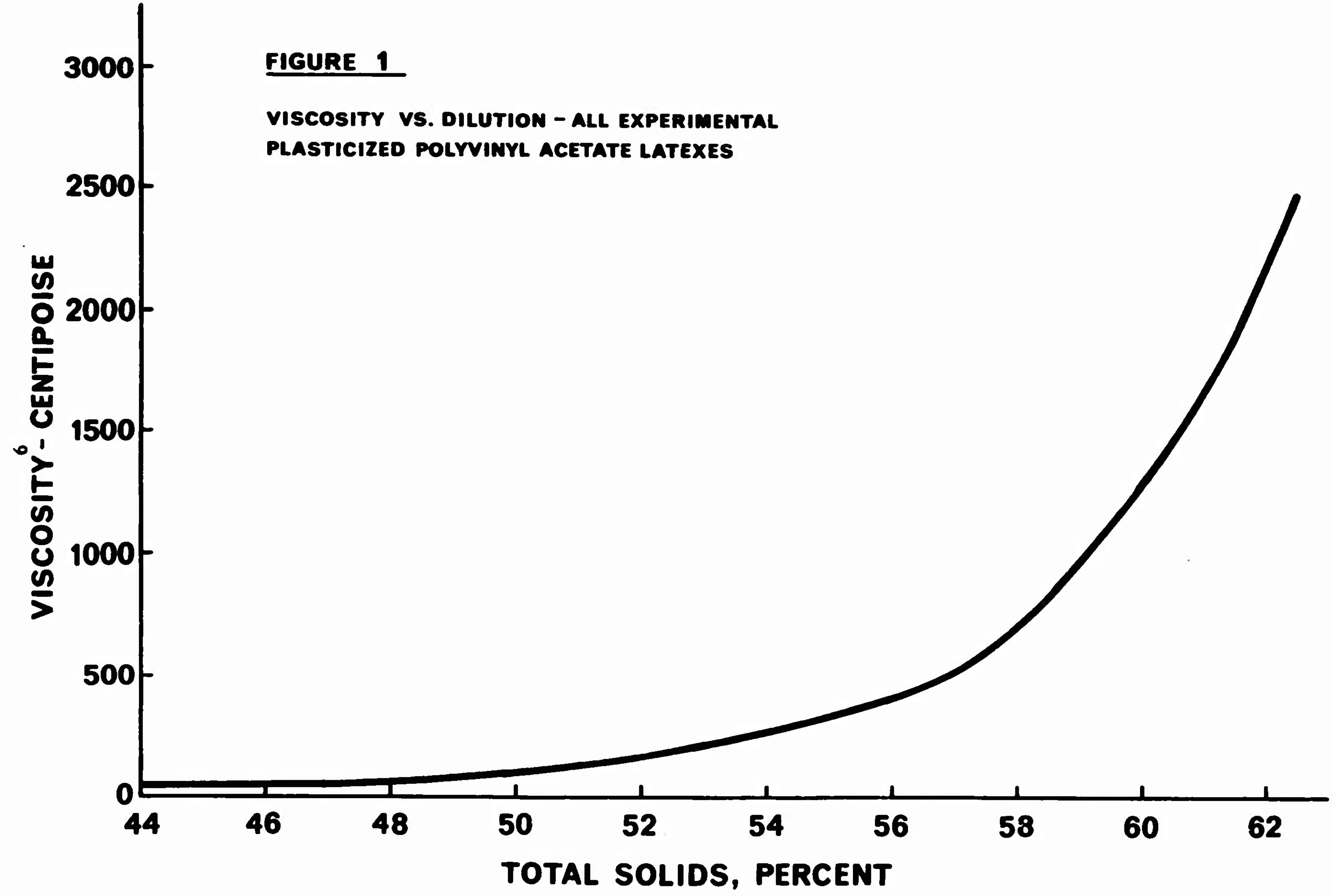


FIGURE 1

VISCOSITY VS. DILUTION - ALL EXPERIMENTAL PLASTICIZED POLYVINYL ACETATE LATEXES

In the preparation of 55-gallon quantities of plasticized products, it is important that thorough mixing be achieved. For field-test evaluation, the WES requested 2-drum quantities of five different formulations. These formulations are numbered 1 through 5 in Table 5. A later request was received for formulation 6 of Table 5. The first group of five formulations and the later formulation all gave tacky surfaces in field testing at the WES. Furthermore, four of the six formulations were eliminated on the basis of other deficient physical properties. Because of the deficiencies noted in the initial samples, additional quantities of the two formulations (formulations 4 and 6 in Table 5) selected for further WES testing were prepared using special care to ensure thorough mixing.

The plasticized mixes were made in an open-top, polyethylene-lined steel drums, Type IRH 449. Agitation was supplied by the vertical, reciprocating motion of a circular plate 11-3/4 inches in diameter and 1/8-inch thick, containing 65, 1/2-inch drilled holes, at a rate of 12, 38-inch strokes per minute. The premixed plasticizers were poured into the drum of latex with the agitator in operation over a three-minute period, and the agitation continued for ten additional minutes. All of the plasticizer appeared mixed at the end of the plasticizer-addition period. The content of the drum was sampled for laboratory analysis, and the drum closed for shipment. However, laboratory film-appearance analysis indicated plasticizer dispersion to be borderline; so the drum of product was mixed again for an hour with a Lightnin' mixer, reclosed, and shipped. Data appearing in Table 5 are on samples cast from the remixed product. The film-appearance analysis consisted of placing 10 g. samples of (1) unmodified polyvinyl acetate precursor, (2) well-mixed formulations prepared in the laboratory, and (3) pilot plant-plasticized mixes into 3-inch by 5-inch Teflon molds. During drying it was noted that mixes prepared in the pilot plant with insufficient agitation exhibited an oily surface appearance in the wet state compared to the other samples. After air-drying for at least 4-6 hours, the poorly mixed pilot-plant samples had a dull surface appearance while the control samples were glossy. After further pilot-plant mixing, as described above, the samples were rerun and all formulations gave the same film appearance as the controls by this test.

I. Manufacturing Procedure - Three-thousand-gallon quantities of each of two formulations were prepared in Union Carbide manufacturing facilities. One formulation contained 10.5 phr. Santicizer 140 and 4.5 phr. FLEXOL 4GO. This product was given the code designation EDCA 1290 by WES. The second formulation, designated EDCA 1295, contained 3.5 phr. Santicizer 140 and 6.5 phr. FLEXOL 4GO.

The mixes were prepared in a 4600-gallon autoclave, agitated at 100 rpm., with the premixed plasticizers added to the precursor latex over a one-hour period. Agitation was provided by a 54 inch Pfaudler-type agitator with three blades of the retreat-blade type. Ambient temperatures were used. Mixing was continued for 30 minutes after addition to ensure homogeneity. The laboratory film appearance analysis test described in the section on pilot-plant production indicated complete incorporation of the plasticizer. If product is to be prepared in any other autoclave or mixing device, the laboratory film appearance analysis test should be used during the mixing procedure to arrive at an adequate mixing cycle.

CONCLUSIONS AND RECOMMENDATIONS

(1) Several plasticizers, much lower in volatility than the dibutyl phthalate used in DCA-70, have been found to be compatible with the polyvinyl acetate latex used in the production of DCA-70. Products based on these plasticizers yield films which retain more flexibility than films made from DCA-70 when subjected to oven aging.

(2) On the basis of the test results obtained in the laboratory and in field trials, the best plasticizer system for an improved version of DCA-70 is a mixture of Santicizer 140 and FLEXOL 4-GO. Two formulations containing these plasticizers but varying in the quantities of each used have been recommended for further field trials.

(3) The substitution of the Santicizer 140-FLEXOL 4-GO combination for dibutyl phthalate does not adversely affect minimum filming temperature, filterable solids, mechanical stability, or aging properties of the latex.

(4) The Santicizer 140-FLEXOL 4-GO formulations require more agitation with the polyvinyl acetate latex during manufacture than does dibutyl phthalate. Without thorough mixing, imbibition of the plasticizer into the latex particles is too slow and the resultant product is deficient in tensile properties when cast into films. A method for assessing the thoroughness of mixing was developed and is described.

(5) Because filling field dispensers is greatly facilitated by decreasing the solids level of the latex, and hence, its viscosity, it is recommended that the solids level of the dust control latex be maintained at about 56%. The lower solids level will also alleviate skinning problems and may improve storage stability.

TABLE 1

PLASTICIZERS SCREENED FOR COMPATIBILITY

Plasticizer	Chemical Name or Formula	Boiling Point °C 5 mm Hg	Vapor Pressure mm Hg at 200°C	Film Forming Property[b]
FLEXOL NODP	n-Octyl, n-Decyl Phthalate	250	---[a]	3°
FLEXOL 10-10	Didecylphthalate	261	0.01 mm	3°
FLEXOL 13-13	Ditridecylphthalate	242 /2mm	0.01 mm	3°
FLEXOL DIOP	Diisooctylphthalate	236	1 mm	3°
FLEXOL TCP	Tricresyl phosphate	241-255 / 4 mm	0.01 mm	2°
FLEXOL PEP	$O(C_6H_8)(COOC_{10}H_{21})_2$	200, decomposes	0.3 mm	3°
FLEXOL A-26	Di-2-ethylhexyladipate	214	2 mm	2°
FLEXOL Z-88	Bis-2-ethylhexyl azelate	237	---[a]	2°
FLEXOL 3-GH	Triethyleneglycol di-2-ethylbutyrate	196	6 mm	1°
FLEXOL 4-GO	Polyethyleneglycol di-2-ethylhexanoate	218	---[a]	1°
Santicizer 214	Mixture of DIOP and aromatic extender	---[a]	---[a]	3°
Santicizer 140	Cresyl diphenyl phosphate	235-255/ 4 mm Hg	0.01 mm	2°
Benzoflex 9-88	Propylene glycol dibenzoate	225-235	---[a]	2°
FLEXOL DBP	Dibutylphthalate	340 /760 mm	0.01 mm at 20°C	1°
FLEXOL TCP + 5 phr Butyl CARBITOL acetate		---[c]	---[c]	1°
Santicizer 140 + 5 phr Butyl CARBITOL acetate		---[c]	---[c]	1°
Benzoflex 9-88 + 5 phr. Butyl CARBITOL acetate		---[c]	---[c]	1°

[a] Data not available

[b] 1° good stong elastic film; 2° moderately good film, slightly brittle; 3° poor film, very brittle

[c] Data not applicable because of mixed system

TABLE 2

FIRST SERIES OF AGING TESTS WITH VARIOUS PLASTICIZERS

Latex Plus Plasticizer	Room Temp. Aging		Thermal Aging, 140°F for 100 hrs.			Static Water Extraction 100 hrs.		Comments
	Tensile psi	Elong. %	Tensile psi	Elong. %	% of Plasticizer lost	Tensile psi	Elong. %	
Santicizer 140 - 10 phr BCA - 2 phr	673	260	1660	170	17.7[b]	---[a]	---[a]	
Santicizer 140 - 10 phr BCA - 5 phr	377	330	1334	170	34.5[b]	---[a]	---[a]	
Santicizer 140 - 15 phr	916	255	980	235	5.1	835	235	Remains pliable after 140°F aging
Santicizer 140 - 20 phr	1044	233	664	260	3.6	1083	248	Remains pliable after 140°F aging
Benzoflex 9-88 - 10 phr BCA - 2 phr	769	258	1453	240	19.5[b]	1973	150	Very brittle after 140°F aging
Benzoflex 9-88 - 10 phr BCA - 5 phr	626	311	1147	240	30.0[b]	2416	210	
FLEXOL 4-GO - 12 phr	453	348	537	336	6.3	---[a]	---[a]	
FLEXOL 3-GH - 8 phr	874	296	1014	230	21.6	1266	185	Brittle after 140°F aging
FLEXOL TCP - 10 phr BCA - 5 phr	408	310	911	80	29.2[b]	---[a]	---[a]	
FLEXOL TCP 10 phr BCA - 2 phr	770	215	1886	220	16.7[b]	---[a]	---[a]	
FLEXOL DBP - 10 - phr (DCA-70 Formulation)	790	255	1113	175	30.0	---[a]	---[a]	

[a] Dashes indicate data not available

[b] Plasticizer weight loss equivalent to BCA present.

TABLE 3
SECOND SERIES OF AGING TESTS WITH VARIOUS PLASTICIZERS

Latex Plus Plasticizer	Latex Lot No.	Room Temp. Aging		Thermal Aging, 140°F for 100 hrs.			Static Water, Extraction, 100 hr.[a]		Minimum Filming Temp.,°F
		Tensile psi	Elong. %	Tensile psi	Elong. %	% of Plast. Lost	Tensile psi	Elong. %	
FLEXOL DBP - 10 phr (DCA-70 Formulation)	2816-A	1381	146	Too Brittle		24.0	---	---	52
FLEXOL 4-GO - 10 phr	2816-A	1606	225	1808	255	5.7	1516	228	51
FLEXOL 4-GO - 12 phr	2816-A	1405	249	1214	257	5.4	---	---	44
Santicizer 140 - 20 phr	2816-A	1148	260	1385	233	2.8	---	---	44
FLEXOL DBP - 10 phr	2821-A	1376	198	Too Brittle		28.2	1282	142	---
FLEXOL 4-GO - 12 phr	2821-A	1011	319	1278	270	5.4	1116	253	---
Santicizer 140 - 20 phr	2821-A	1205	250	1358	198	2.8	---	---	---
Santicizer 140 - 14 phr FLEXOL 4-GO - 6 phr	2821-A	835	291	982	318	3.4	---	---	---
Santicizer 140 - 10 phr FLEXOL 4-GO - 4.5 phr	2821-A	1398	267	1531	228	4.7	1420	242	---
Santicizer 140 - 5 phr FLEXOL 4-GO - 5 phr	2821-A	1409	170	893	180	6.4	1400	138	---

[a] Dashes indicate data not available

TABLE 4

THIRD SERIES OF AGING TESTS WITH VARIOUS PLASTICIZERS

Latex Plus Plasticizer	Room Temp. Aging		Thermal Aging						Running Water Extraction 340 hrs.	
			140 °F for 170 hrs			140 °F for 340 hrs.				
	Tensile psi	Elong. %	Tensile psi	Elong. %	% of Plast. Lost	Tensile psi	Elong. %	% of Plast. Lost	Tensile psi	Elong. %
FLEXOL 4-GO - 12 phr	464	388	648	382	5.7	515	300	12.8	703	283
FLEXOL 4-GO - 10 phr	791	375	722	330	7.7	678	324	14.3	744	277
Santicizer 140 - 20 phr	473	300	596	318	4.2	574	295	6.4	---[a]	---[a]
Santicizer 140 - 10.5 phr FLEXOL 4-GO - 4.5 phr	727	400	680	338	5.2	566	296	9.1	514	276
Santicizer 140 - 3.5 phr FLEXOL 4-GO - 6.5 phr	830	297	---[a]	---[a]	---[a]	863	272	11.3	863	250
FLEXOL DBP - 10 phr (DCA-70 formulation)	590	352	1011	220	50.0	1048	174	61.0	650	245

[a] Dashes indicate data not available

TABLE 5

TENSILE DATA AND AGING TESTS ON MATERIAL USED IN WES FIELD APPLICATION

			Aged 7 days at room temperature		Aged for 107 hours at 140°F.		
Formulation	Plasticizer	phr	Tensile psi	Elong. %	Tensile psi	Elong. %	% of Plast. Lost
1	FLEXOL 4-GO	12	1073	298	1446	288	6.8
2	FLEXOL 4-GO	10	1287	267	1620	256	6.3
3	Santicizer 140	20	1039	233	1600	220	4.1
4	Santicizer 140 FLEXOL 4-GO	10.5 4.5	1343	256	1517	238	4.9
5	FLEXOL 4-GO Santicizer 140	2.5 4.0	1527	229	1769	203	6.5
6	Santicizer 140 FLEXOL 4-GO	3.5 6.5	1585	220	2000	220	6.5

TABLE 6

AGING STUDIES ON POLYVINYL ACETATE LATEX COMPOUNDED WITH VARIOUS PLASTICIZERS

Latex Plus Plasticizer	Aging Conditions	Viscosity * Centipoise	Filterable Solids on 60 mesh - %	pH
Original Latex No Plasticizer	1 day at 25 °C	1630	0.0106	4.40
FLEXOL 4-GO - 10 phr	↓	2200	0.0160	4.35
FLEXOL 4-GO - 20 phr	↓	2600	0.0103	4.40
Santicizer 140 - 20 phr	↓	2800	0.0180	4.40
Santicizer 140 - 40 phr	↓	5050	0.0670	4.40
Santicizer 140 - 10.5 phr FLEXOL 4-GO - 4.5 phr	↓	2650	0.0385	4.35
Santicizer 140 - 21.0 phr FLEXOL 4-GO - 9.0 phr	↓	4500	0.0420	4.40
FLEXOL 4-GO - 10 phr	2 wks. at 25 °C	2400	0.0143	4.35
FLEXOL 4-GO - 20 phr	↓	3050	0.0235	4.35
Santicizer 140 - 20 phr	↓	3050	0.0192	4.40
Santicizer 140 - 40 phr	↓	6850	0.0260	4.40
Santicizer 140 - 10.5 phr FLEXOL 4-GO - 4.5 phr	↓	2680	0.0170	4.40
Santicizer 140 - 21.0 phr FLEXOL 4-GO - 9.0 phr	↓	4930	0.0218	4.35
FLEXOL 4-GO - 10 phr	2 wks. at 50 °C	1900	0.0174	4.15
FLEXOL 4-GO - 20 phr	↓	2600	0.0150	4.05
Santicizer 140 - 20 phr	↓	2150	0.0210	4.10
Santicizer 140 - 40 phr	↓	3950	0.0460	4.05
Santicizer 140 - 10.5 phr FLEXOL 4-GO - 4.5 phr	↓	1900	0.0185	4.10
Santicizer 140 - 21.0 phr FLEXOL 4-GO - 9.0 phr	↓	4000	0.0330	4.10

* Brookfield No. 4 spindle LVT at 60 rpm.

DEVELOPMENT OF PROCUREMENT SPECIFICATIONS FOR AN IMPROVED DUST-CONTROL SYSTEM BASED ON POLYVINYL ACETATE LATEX (PHASE II)

INTRODUCTION

The primary purpose of procurement specifications is to permit the end-user to select the product needed for his requirements using relatively simple laboratory procedures instead of more elaborate and expensive end-use tests, to establish the quality of the product, and to ensure proper performance in the intended application. Inherent in this statement is the assumption that suitable laboratory tests can be devised to measure those quality factors which affect end-use performance. Fortunately, many tests have been developed which measure the fundamental properties of latex products. In the work reported here, these tests have been applied to polyvinyl acetate latexes from many commercial sources. Correlating the test results with what is known about the behavior of latexes in the dust-control application allows one to set standards based on fundamental tests which should ensure proper performance. However, it must be kept in mind that specifications may be expected to evolve with experience. It may be advisable to modify test methods, specification values, etc., as experience is gained.

Wherever possible, test methods used and described in the report are ASTM procedures or modifications thereof. Some of the test methods used were described in the report on Phase I of the project. However, generally speaking, the test work on Phase I did not require precise results since this effort was aimed at determining feasibility rather than at precision. Consequently, despite the resulting redundancy, it was felt advisable to repeat the description of the test methods, adding all necessary details required to ensure good test precision.

EXPERIMENTAL PROCEDURES

A. Film Preparation - In order to ensure good precision of tensile strength and elongation tests it is necessary to use carefully controlled laboratory techniques in the casting and handling procedures. Unfortunately, it is not possible to prescribe a single technique. Latex products vary in viscosity and the procedure for a high viscosity latex requires modification for lower viscosity products. High viscosity materials can be spread with a doctor blade on a glass substrate without encountering thinning due to run-off. For low viscosity latexes, a mold of silicone rubber or a Teflon-coated metal mold must be used. When casting on glass, a fluorocarbon release agent (MS-122 made by Miller-Stephenson Chemical Company or its equivalent) should be sprayed on the glass and allowed to dry before spreading the latex.

The procedure for latexes which do not thin out when spread on glass was as follows:

1. Stir the latex to be cast in order to ensure homogeneity.

2. De-aerate the sample by centrifuging, or alternatively use the procedure of ASTM D 1417 sections 7.2.1 and 7.2.2.

3. Prepare a plate glass surface by spraying with a fluorocarbon release agent.

4. Select a doctor blade applicator with a clearance such that a dry film thickness of 0.0100± 0.0010 inches is obtained.

5. Place the doctor blade on the prepared plate glass surface and pour the material to be cast in the trough of the blade.

6. Pull the doctor blade at a constant speed across the glass surface.

7. Clean the doctor blade after each film draw-down operation.

8. Films should be permitted to dry at least three hours (until the film becomes transparent) at room temperature on a level surface free from drafts. (See ASTM D 618 section 3c) .

9. The film is then removed from the glass substrate and hung to dry at room temperature for a total of 24 hours from the time of casting.

10. Proceed to cure films as specified in individual tests.

For latexes which spread out on glass, the procedure was modified by using a Teflon mold with a flat bottom and raised edges so as to provide a contained area the size of the finished film. Latex in sufficient quantity to yield a 0.010 inch film is carefully poured into the mold. Drying and conditioning steps are the same as indicated above.

B. Tensile Strength and Elongation - ASTM Method D 822, "Tensile Properties of Thin Plastic Sheeting", was used with minor modifications. The constant rate-of-grip separation procedure (Method A, section 1b) was employed. A cutting die of Type B described in ASTM D 412 was used to prepare dogbone specimens (See Section 4d of D 822) . Since the conditioning procedure used on film test specimens made from polyvinyl acetate affects their tensile properties, a more elaborate procedure than described in sections 4e and 4f of D 822 was adopted. The films were aged under laboratory conditions for 24 hours after casting and then placed in a 23 °C, 50% relative-humidity room for 144 hours prior to tensile testing. Dogbone specimens were cut 24 hours prior to testing. Five dogbones were cut from each film sample. An initial straining rate of 10 inches per inch per minute was selected, an extensometer was employed, and air-activated grips were used to minimize slippage. Tensile strength and elongation at break were determined for each of the five specimens from each film sample.

Each of the five specimens normally gives different values for tensile strength and elongation. Some type of averaging procedure must be employed in order to assign a single value for each of these parameters to the film sample. While the arithmetic mean has often been used, there are cogent arguments against this simple averaging procedure. Abnormally low values for tensile strength and elongation are often obtained on certain specimens due to inadvertent flaws of one type or another. Low results of this type can unduly influence the mean. ASTM Method D 412 suggests selecting the test result for the specimen which yields a result intermediate between the two high and two low results. This procedure was adopted.

C. Total Solids - Proper sampling of a batch is required to obtain reproducible results. Section 2 of Method D 1417-67 entitled "Standard Methods of Testing Synthetic Rubber Latices" describes a good procedure for obtaining representative samples.

Total solids determinations were done by method ASTM D 1417-67, section 3. Disposable aluminum liners for samples were used for convenience. A drying time of one hour was found to be sufficient to effect complete removal of water.

If it appeared that complete drying was not obtained in one hour, the sample was replaced in the oven as many times as necessary to achieve constant weight.

D. Surface Tension - The measured value of surface tension is dependent on the total solids content of the latex. When a procurement specification is established, there will be a specification on total solids, thus obviating the need for dilution to a standard total solids value. However, surface tensions can be determined more precisely at lower viscosity values and hence, the specification method calls for diluting two parts of latex with one part distilled water prior to measurement. Surface tension values quoted in this report were obtained at two levels of total solids. If the as-received latex was higher in total solids than called for in the specification, it was diluted to the standard value and the surface tension measured. The sample was then further diluted two parts latex to one part distilled water and a second reading obtained. ASTM D 1417-67, Section 6, outlines the surface tension test procedure.

E. pH - Material for pH determination is to be collected in the same manner as referred to in the preceding section on total solids determination. Section 5 of ASTM D 1417-67 was the procedure followed with two modifications. For section 5.1 on apparatus, a glass electrode with a range suitable for the material to be tested was employed. A Beckman pH meter, Model G, was used in the tests reported here. With regard to section 5.2 on standard solutions, a reference solution with a pH within the specification range for the latex was used. Special care was taken to maintain clean electrodes.

F. Viscosity - The Brookfield viscometer is generally accepted as being most suitable for measuring the viscosity of opaque latex materials. Data obtained in this report were obtained on a Brookfield Synchro-Lectric Viscometer, LV Model. The viscosity was determined using the following combinations of viscosity, spindle size, and spindle rotation speed.

Viscosity, cps.	Spindle No.	Spindle Speed, rpm.
0-80	1	60
80-800	2	30
800-3000	3	30
3000-8000	4	60
8000-30,000	4	30

Preparation of the sample followed the procedure of ASTM D 1417 sections 7.2 and 7.3. Instead of the 25° ± 2°C temperature recommended in section 7.3, 20° ± 1°C was employed. The lower temperature delays the formation of film on the latex surface during viscosity measurements.

Since the recommended specification range for viscosity (see Section 3.8) is 800-3000, the specification test method requires only the No. 3 spindle at 30 rpm.

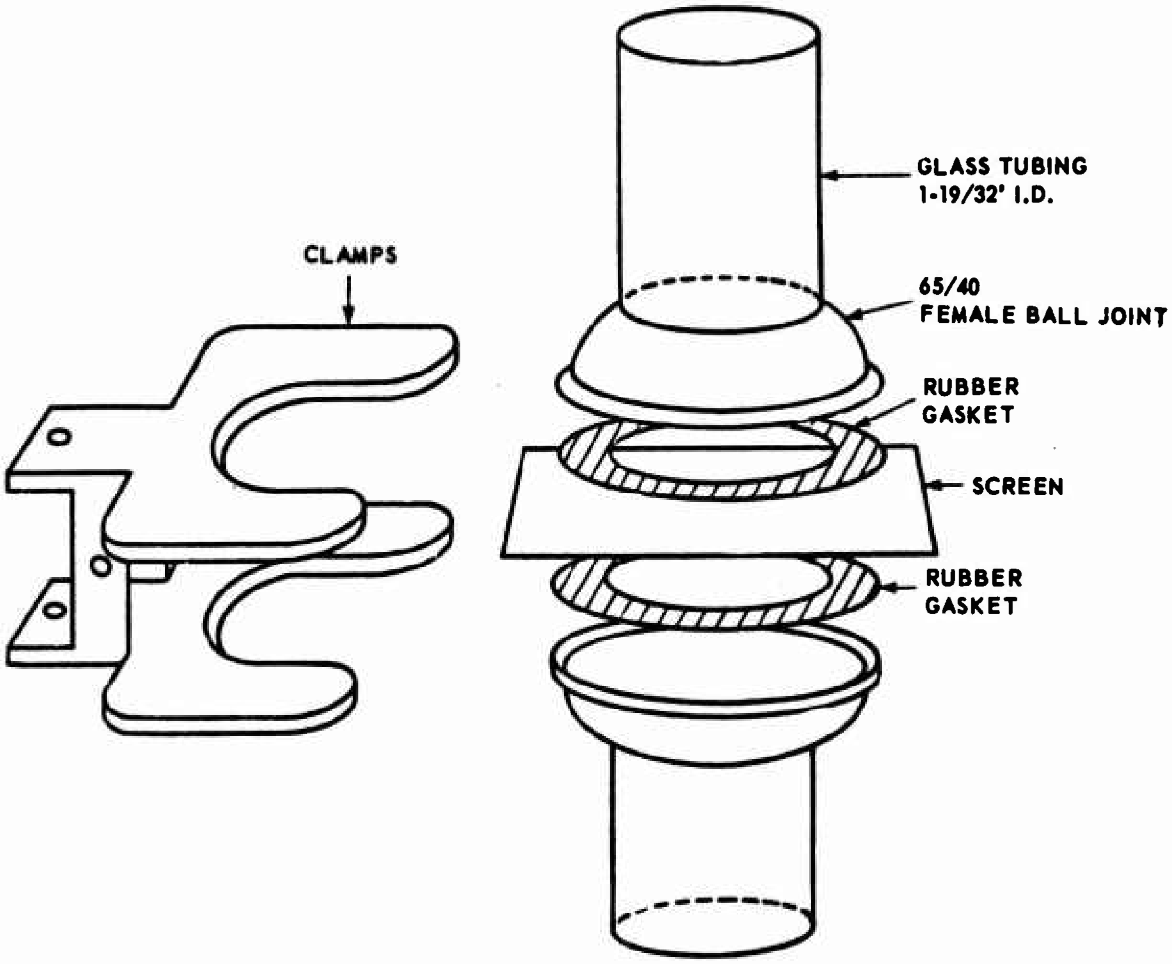
G. Filterable Solids - The material to be tested was sampled using the same procedure as given under the section on total solids determination. In order to calculate the filterable solids value the total solids content must also be known or determined. To determine filterable solids, an approximately one thousand gram sample of the latex was weighed out and the weight recorded to the nearest 0.1 gram. The sample was diluted with one to three times its volume of cold tap water and stirred to obtain a homogeneous dispersion. In sampling the latex any surface film which had formed due to excessive air space in the container in which it was stored was removed from the sample.

The measurement of filterable solids utilized the apparatus shown in Figure 2. The 60-mesh stainless steel screen with wire size of 0.0075" was weighed to the nearest milligram. The diluted latex sample was then poured through the screen after the screen had been inserted in the apparatus described in Figure 2. Immediately after all the sample had passed through the screen, the collected solids on the screen were washed with a gentle stream of cold tap water until the water passing through the screen was clear.

The screen was then placed in an oven at 135° ± 1°C and dried to constant weight. The screens were weighed to the nearest milligram.

Filterable solids were calculated by multiplying the weight of the sample by the percent total solids. This figure was divided into the difference between the weight of the screen after the test and the tare weight. Multiplying this figure by 10^4 gives filterable solids, percent by weight.

FIGURE 2 - SCREEN ASSEMBLY FOR FILTERABLE SOLIDS DETERMINATION



H. Salt Tolerance - This test was carried out with the same apparatus as used in the filterable solids test. Five-percent solutions of borax and sodium chloride were prepared in quantities of 100 ml. each and these solutions were used to dilute the latex instead of the tap water employed in the filterable solids test. In the salt tolerance test, a 1:1 dilution should not be exceeded. After the sample was poured through the screen, cold tap water was used to wash the screen and the remainder of the procedure was the same as for the filterable solids test.

I. Storage Stability at 140°F - An oven capable of holding a temperature of 140 ± 2°F was used. Two wide-mouth jars, approximately 1-3/4" diameter by 4-1/4" high with screw-cap covers fitted with noncorroding liners were used. If a substitute jar is used, it must be capable of holding 180 ± 30 ml. of sample.

The latex was sampled as previously described. Two jars were filled with samples from each individual lot. The jars are filled to within one-eighth inch of the top.

One jar was stored at standard laboratory conditions and the other jar was placed in the oven at 140°F for 240 hours. The oven sample was removed after the test period and permitted to return to standard laboratory temperature before removing the cover. Any surface film that had formed was removed. Films were prepared from both the oven-heated and the control sample using the method previously described. Tensile strength and elongation were determined on the cast film after curing.

J. Shear Stability - The shear stability of the latex is an important property because any degradation or coalescence during pumping could result in clogged equipment or a final film deficient in physical properties. Published tests in the literature for latex materials are not sufficiently rigorous to ensure proper performance. Consequently, a proposed ASTM test designed for measuring shear stability of polymer-containing oils was adopted for this study. The ASTM test method has not yet been formally adopted. but it is described in Appendix XII, "ASTM Standards on Petroleum Products and Lubricants", October 1961, Vol. 1, p. 1160, and is entitled, "ASTM Proposed Method of Test for Shear Stability of Polymer-Containing Oils".

The apparatus used was a Raytheon Magnetostrictive Oscillator, Model DF 101. A reference fluid conforming to specification MIL-H-5606 BC 10.1572 was used to calibrate the apparatus. A kinematic viscometer tube number 200-H-259 was employed to measure the viscosity of the sample. The instrument settings were adjusted to yield a 15% decrease in viscosity of the reference fluid when 30 ml. of sample was sheared for 30 minutes.

The plasticized polyvinyl acetate latexes were tested using the ASTM procedure but with three changes. The sample size was increased to 100 ml, the circulating water temperature was set at 70 ± 5 °F, and the viscosities of the latex were run with a Brookfield viscometer as described previously.

TEST RESULTS

A. Effect of Latex Production Lot on Tensile Strength and Elongation - Five separate lots of the polyvinyl acetate latex used in the preparation of DCA-70 were procured from Union Carbide manufacturing facilities and each of the lots was plasticized using three different formulations. In one case FLEXOL 4-GO at 6.5 phr (phr is parts per 100 parts resin) and Santicizer 140 at 3.5 phr was used, the second employed FLEXOL 4-GO at 4.5 phr and Santicizer 140 at 10.5 phr, while the third formulation used Santicizer 140 at 20 phr.

Tensile strengths and elongations are recorded in Table 7. It will be noted that the tensile strength decreases with increasing plasticizer concentration. While differences are noted in the tensile strengths measured for the various manufacturing lots plasticized according to a given formulation, these differences appear to be within experimental error. It is significant to note that no single lot gives consistently high or low results with each of the three formulations.

B. Precision of Tensile Strength and Elongation Tests - Twenty replicate films were cast from each of several plasticized formulations for determination of the confidence limits on the tensile strength and elongation tests. A single lot of polyvinyl acetate latex precursor was plasticized using the same three formulations described in the previous section A under Test Results. The total solids content of the precursor latex was 60%. Films were prepared in two ways: (1) using the plasticized latex without water dilution, and (2) diluting the plasticized latex with water to bring the solids level to 57% for the 6.5 phr FLEXOL 4-GO, 3.5 phr Santicizer 140 formulation; 56% for the 4.5 phr FLEXOL 4-GO, 10.5 phr Santicizer 140 formulation; and 55% for the 20 phr Santicizer 140 formulation.

Calculated results on 95% and 99% confidence limits for single measurements of tensile strength and elongation are recorded in Table 8. The most pertinent observation to be made is that test precision is superior for the samples prepared from latexes which had been diluted with water. Since there are other advantages of lower solids content (e.g. lower viscosity, easier dispenser filling, less skinning, etc.), the product will be recommended to contain total solids in the 55-57% range. (See Section C under Recommendations).

C. Effect of Relative Humidity on Tensile Strength and Elongation - Strength test results on polyvinyl acetate films are strongly affected by moisture content due to pickup from atmospheric humidity. Absolute control of relative humidity in testing laboratories subject to variations in occupancy, etc., is difficult. In all the tensile testing reported here, strenuous attempts were made to ensure constant humidity conditions, but it is not certain that this was always achieved. Some attempts were also made to ascertain the effect of relative humidity on strength; erratic results were obtained and the data are not reported.

D. Tensile Strength and Elongation of Various Commercial Polyvinyl Acetate Latexes after Plasticization - Commercial polyvinyl acetate latexes were obtained from various sources (Commercial products are identified by code letters in the tabulated data. Identification of the corresponding trade names and suppliers is given in Table 9.) and plasticized using the same three formulations described in previous sections. Films were cast and tensile properties determined. Most of these latexes gave films of very poor strength. The films actually cracked and broke either during the seven-day conditioning period or while attempting to die-cut tensile specimens. Data obtained on tensile strength and elongation are recorded in Table 10.

It is quite obvious that very few plasticized polyvinyl acetate latexes yield films of sufficient strength to be usable in dust control applications. The tensile test clearly indicates that only two of the precursor latexes give high strengths. Hence, a specification on tensile strength and elongation of cast films is desirable to eliminate unsatisfactory polyvinyl acetate latex products from consideration.

E. Total Solids - Values for contained total solids of commercial polyvinyl acetate latexes after plasticization are recorded in Table 11.

F. Surface Tension - Data obtained on commercially available polyvinyl acetate latexes plasticized with dibutyl phthalate or with the three formulations developed in Phase I of this contract are given in Table 11. Surface tension data were obtained on the latex as received but after plasticization, and after dilution with distilled water at a ratio of two parts latex by volume to one part water. Total solids values on the plasticized latexes and pH values are also recorded in Table 11.

Most of the latex products have reasonably high surface tensions. Those latexes with lower values of surface tension tend to foam when agitated mechanically, for example, in the shear stability test. Since foam must be avoided during latex pumping in field application, a specification minimum for surface tension appears advisable.

G. pH - Values for pH of various commercial polyvinyl acetate latexes after plasticization are given in Table 11.

H. Viscosity- Data are given in Table 11.

I. Filterable Solids - All twelve polyvinyl acetate precursor latexes were tested for filterable solids. Materials plasticized by the manufacturer were tested as received. The other latexes were plasticized using the three formulations previously referred to.

Data obtained are recorded in Table 12. It will be noted that all the latexes increase in filterable solids on addition of plasticizer. Generally, the filterable solids level on the precursor polyvinyl acetate latexes is 0.01% or less.

J. Salt Tolerance - The commercial latex products were subjected to the salt tolerance test described in the section on experimental procedures. None of these materials coagulated due to the salt, and filterable solids obtained when borax or sodium chloride solutions were employed to dilute the sample were essentially identical to filterable solids obtained on dilution with tap water. Because of the lack of any effect, the data are not recorded in this report. This result is not surprising because the commercial latex products selected for this study were all of types expected to possess good salt tolerance.

K. Storage Stability at 140° F - Although it is not recommended that latexes be stored at temperatures over 125° F for any length of time, it would appear advisable that a material intended for military usage be capable of withstanding moderately high temperature storage. All latex materials formed a surface skin on prolonged heating at 140 °F. However, the material beneath the skin generally remained usable.

The storage stability test was carried out with the polyvinyl acetate latex used in the production of DCA-70. It was plasticized using the three formulations previously described. Data obtained are recorded in Table 13.

L. Shear Stability - Shearing the latex samples may effect changes in viscosity or in filterable solids level. Also, certain latex products foam excessively. Any of these changes could affect the performance of the product in its field application. The commercially available polyvinyl acetate latex products were tested after plasticization to ascertain the magnitude of these effects. Data are recorded in Table 14.

It will be noted that most of the latexes did not change much in filterable solids level. Some, in fact, decreased--probably due to break-up of seeds in the latex by shearing action. Changes in viscosity are more significant, at least in some of the products. The change in viscosity is sufficient for some products to suggest they may not perform adequately. The foaming tendency noted for one latex product is bad. Generation of foam in distributor equipment would be a considerable handicap to proper application.

RECOMMENDATIONS

This section of the report proposes an interim specification scheme to ensure adequate performance of products procured for dust-control applications. As experience is accumulated, it should be possible to modify, simply, and augment the specification proposals. It is vital that products intended for this use perform as expected and initially, one should err on the side of tighter specifications to ensure adequate performance. If experience and economics suggest less stringency is allowable, suitable modifications can be made.

A. Composition - The supplier shall certify that the product submitted is a homopolymer latex of polyvinyl acetate. All components of the latex shall be essentially non-toxic. No chemicals known to cause allergenic reactions or to give off noxious or harmful vapors shall be used. It shall be formulated with the plasticizer selected and the quantities specified. Unless further experience leads to a different selection, the formulation will be FLEXOL 4-GO at 6.5 phr and Santicizer 140 at 3.5 phr.

B. Tensile Strength and Elongation - These parameters are probably the two most important properties ensuring adequate product performance in the dust-control application. It is difficult to question the premise that the film on the soil surface must be strong for the intended application. Unfortunately, because of the inherent effects of flaws in test specimens and the effect of relative humidity, tensile strength and elongation are difficult to measure with high precision.

To ensure adequate performance of submitted products, it is proposed that all candidate materials first be subjected to a qualification procedure. The manufacturer seeking qualification shall submit samples from two production lots of the product in question. Ten separate films shall be cast from each production lot. At the same time, ten films shall be cast from a plasticized latex previously accepted for dust-control use. The thirty films shall be conditioned simultaneously and tensile strength tests run with random sequential selection of test specimens from the 150 dogbones.

A statistical analysis shall then be carried out on the three groups of tensile and elongation measurements. The Student t test (see any standard text on statistical methods) shall be used to ascertain whether any significant differences exist among the three groups. If the statistical analyses show that, at the 95% confidence level, one or both of the submitted lots of latex yields either a significantly lower or higher mean tensile strength or elongation than the standard, the product is rejected. If no significant differences exist, the product is approved.

One might question whether a submitted product yielding significantly higher values for both tensile strength and elongation than the control should be rejected. This course of action is recommended because a significant difference of this type suggests a product of an essentially different type has been achieved. It may actually be a better product for the dust-control application than the previously approved control. However, in the unlikely case that these circumstances are encountered, it would appear advisable to proceed cautiously, with field evaluations and other testing to ensure acceptable performance of the product in all respects.

For products that have previously qualified, tensile strength and elongation shall be tested on each production lot. The data of Table 10 provide the information needed to set specifications. Random test variation on tensile strength of products made to formulation A (6.5 phr FLEXOL 4-GO, 3.5 phr Santicizer 140) should yield 19 out of 20 results in the range of 1326 to 1724 psi. A production lot yielding a tensile strength of 1326 psi or over is accepted. If the film yields a tensile value of less than 1326 psi, a second film may be prepared and tensile tests run. If it yields a value less than 1326 psi, the product is rejected. If 1326 psi or over, the tensile test result on the first film is rejected, and the product is accepted. This go-no-go test sequence is advisable to allow for those statistical variations in the test which may result in suitable materials being rejected.

A similar procedure is suggested for elongation results. In this case with formulation A, the specification elongation is a minimum of 226%.

C. Total Solids - A specification on total solids content is advisable simply because the concentration of the active ingredient in the dust-control latex should be controlled. DCA-70 itself contains about 60% solids and field experience indicates this level can be handled albeit with some difficulties because the viscosity at this solids level is quite high and this leads to problems in transferring the material from drums to the distributing equipment. Higher solids contents also cause more problems with surface skinning, etc. The experimental latexes developed in Phase I were made in quantity at solids levels lower than 60%. These products were much more readily transferrable from drums to dispensing equipment. Consequently, the solids level specification should be: Total solids = 55 to 57%.

D. Surface Tension - A specification for surface tension of the dust-control latex should be imposed because this is an indirect method of controlling the foaming tendency of the product. According to Table 11, DCA-70 has a surface tension of 43.5 dynes/cm. after dilution. When the same polyvinyl acetate latex used in manufacturing DCA-70 was plasticized according to the three formulations under consideration, the surface tension values after dilution were 43.8, 44.2 and 48.2 dynes/cm. Without field experience on the relationship between surface tension and foaming tendency, it is difficult to suggest a minimum value for surface tension which will ensure proper performance. Until such experience is gained, it is suggested that a minimum of 40.0 dynes/cm. be imposed.

E. pH - The acid or basic properties of a latex have an indirect effect on its utility because materials of construction not subject to corrosion must be utilized in all dispenser parts which contact the latex. A specification range of 4.0 to 6.0 is recommended. Most latexes tested (Table 11) are in the 4.0 to 5.0 pH range but inclusion of less acidic materials seems desirable because such products would be less corrosive.

F. Viscosity - In the application of a dust-control latex, filling the dispenser is facilitated if the viscosity of the product is on the low side. It is not expected that viscosity would have any other effect on the behavior of the product. A specification range of 800 to 3000 cps is recommended. As pointed out in the section on test methods, the viscosity is to be measured at 20° ± 1°C. with a No. 3 spindle at 30 rpm.

G. Filterable Solids - It does not appear that filterable solids level is as critical for the dust-control application as it would be for many other applications of polyvinyl acetate latexes. Only one sample of the many tested gave a value of over 0.03%. A specification of 0.03% maximum filterable solids seems reasonable.

H. Salt Tolerance - This parameter is of some importance because it is conceivable that one may need to use brackish water to dilute the dust-control latex in field applications. As pointed out previously, all the latex products investigated are of types which have excellent salt tolerance. However, many latex products are deficient in this property and, to ensure proper performance, any specification scheme should eliminate these products from consideration.

Setting specification values should not be difficult. Latex products which fail the salt tolerance test generally coagulate completely on addition of salt. Hence it should be sufficient to specify that the filterable solids level in the salt tolerance test shall not be greater than 0.1%.

I. Storage Stability at 140°F - Latex products which fail to withstand this test will in all likelihood deteriorate so badly that it will not be possible to cast a coherent film after 140°F aging. For specification purposes it is reasonable to suggest the aged sample, when cast into film, shall retain at least 80% of the tensile strength and elongation of the control sample.

J. Shear Stability - This parameter is of great importance because the facility with which the product can be applied to soil under combat conditions is vital in determining the utility of the product. If possible, difficulties encountered during pumping or dispensing due to coagulation or foaming should be avoided. While the importance of good shear stability is recognized, one must admit that, with the data so far collected, it is the most difficult parameter of those investigated to recommend numerical specification limits. It is suggested that the viscosity should show no greater change than 50% on subjection of the latex to the shear stability test; the filterable solids shall not increase to a level above 0.03%; and the volume of the sample due to foaming shall not be greater than 120% of the initial volume. These limits can be modified as experience is gained.

K. Soil Binding Properties - None of the foregoing tests measures the properties in a closely use-related application. It is entirely conceivable that a latex could meet all the suggested specifications and yet be inadequate in its adhesion to soil. Therefore, the screening tests used by WES for laboratory evaluation of dust-control materials should be retained. A candidate product must qualify for the application by passing all the tests described in the WES procedures, (Appendix A). The qualification procedure will require the testing of two separate production lots of the candidate latex in a manner similar to the proposed qualification procedure given in Section B under Recommendations.

L. <u>Summarized Recommended Specifications</u> - Assuming the formulation to be selected will contain FLEXOL 4-GO at 6.5 phr and Santicizer 140 at 3.5 phr, the recommended specifications are summarized in Table 15.

TABLE 7

EFFECT OF POLYVINYL ACETATE MANUFACTURING LOT ON TENSILE STRENGTH AND ELONGATION OF PLASTICIZED PRODUCTS

Latex Lot No.	FLEXOL 4-GO (6. 5 phr) - Santicizer 140 (3. 5 phr) Tensile Strength, psi.	FLEXOL 4-GO (6. 5 phr) - Santicizer 140 (3. 5 phr) Elongation %	FLEXOL 4-GO (4. 5 phr) - Santicizer 140 (10. 5 phr) Tensile Strength, psi	FLEXOL 4-GO (4. 5 phr) - Santicizer 140 (10. 5 phr) Elongation %	Santicizer 140 (20 phr) Tensile Strength, psi	Santicizer 140 (20 phr) Elongation %
2965	1943	280	1344	260	1183	280
2970	1768	260	1340	290	1271	270
2972A	1860	260	1327	250	1120	260
2973	1880	250	1357	270	1200	230
2974	1806	250	1402	230	1164	270

TABLE 8

CONFIDENCE LIMITS FOR TENSILE STRENGTH AND ELONGATION TESTS

		Tensile Strength, psi			Elongation, %		
Formulation	Total Solids, %	Mean	Confidence Limits, 95%	Confidence Limits, 99%	Mean	Confidence Limits, 95%	Confidence Limits, 99%
A[1]	As mixed	1720	±270	±309	250	±47	±54
B[1]	As mixed	1280	±270	±309	280	±47	±54
C[1]	As mixed	1080	±270	±309	280	±47	±54
A	57	1525	±199	±230	260	±34	±40
B	56	1145	±199	±230	290	±34	±40
C	55	1030	±199	±230	300	±34	±40

[1] Formulation A: 6.5 phr FLEXOL 4-GO, 3.5 phr Santicizer 140

Formulation B: 10.5 phr FLEXOL 4-GO, 4.5 phr Santicizer 140

Formulation C: 20 phr Santicizer 140

TABLE 9

KEY TO CODE DESIGNATIONS OF COMMERCIAL POLYVINYL ACETATE LATEXES

Code Letter	Commercial Name	Supplier
A	DCA-70	Union Carbide Corporation
B	Amsco 3001-P	American Mineral Spirits Co.
C	Amsco 3001	American Mineral Spirits Co.
D	Daratak SP 1011	W. R. Grace
E	Elvacet 81-900	DuPont
F	Vinac WR 50	Airco Chemicals and Plastics
G	Resyn 25-1040	National Starch
H	Polyco 577 G	Borden Chemical Company
I	Wallpol 40-307	Reichhold Chemicals
J	DCA-70 Precursor [1]	Union Carbide Corporation
K	Vinac CE-1-P	Airco Chemicals and Plastics

[1] The polyvinyl acetate latex to which dibutyl phthalate is added to make DCA-70.

TABLE 10

TENSILE STRENGTH AND ELONGATION OF FILMS CAST FROM PLASTICIZED SAMPLES OF COMMERCIAL POLYVINYL ACETATE LATEXES

Latex[1]	Plasticizer[2]	Tensile Strength, psi	Elongation %	Remarks
A	1	1380	145	--
B	1	--	--	Too Brittle
C	2	--	--	Too Brittle
D	2	900	150	--
E	2	--	--	Too Brittle
F	2	--	--	Too Brittle
G	2	1470	290	--
H	2	1156	240	--
I	2	--	--	Too Brittle
J	2	1550	260	--
C	3	--	--	Too Brittle
D	3	1070	180	--
E	3	--	--	Too Brittle
F	3	--	--	Too Brittle
G	3	1275	310	--
H	3	625	300	--
I	3	--	--	Too Brittle
J	3	1150	290	--
C	4	--	--	Too Brittle
D	4	1020	180	--
E	4	--	--	Too Brittle
F	4	--	--	Too Brittle
G	4	1300	330	--
H	4	850	290	--
I	4	--	--	Too Brittle
J	4	1030	300	--
K	[3]	--	--	Too Brittle

[1] Code identification of trade name and supplier in Table 9

[2] Plasticizer identification
1 = 10 phr dibutyl phthalate
2 = 6.5 phr FLEXOL 4-GO
3.5 phr Santicizer 140
3 = 4.5 phr FLEXOL 4-GO
10.5 phr Santicizer 140
4 = 20 phr Santicizer 140

[3] Identity of plasticizer not disclosed by manufacturer

TABLE 11

SURFACE TENSION, TOTAL SOLIDS, AND pH VALUES OF PLASTICIZED COMMERCIAL POLYVINYL ACETATE LATEXES

Latex [1]	Plasticizer[2]	Total Solids, %	Surface Tension, Dynes/cm. As Is	Surface Tension, Dynes/cm. 2:1 Dilution	pH	Viscosity cps
A	1	60	47.8	43.5	4.31	--
B	1	60	47.5	42.7	4.55	2900
C	2	60	45.6	41.0	4.60	2400
D	2	57	51.4	41.2	[4]	6300
E	2	57	41.4	39.7	4.10	2100
F	2	57	44.1	38.8	4.60	2660
G	2	57	34.1	32.4	3.92	1380
H	2	57	[4]	[4]	4.41	1010
I	2	57	39.8	35.1	4.65	1880
J	2	57	46.5	43.8	4.31	1200
C	3	61	45.9	39.6	4.60	2420
D	3	58	43.1	42.6	[4]	7650
E	3	58	42.9	40.7	4.10	2180
F	3	58	45.4	39.3	4.60	2425
G	3	56	36.7	35.8	3.92	1035
H	3	58	[4]	[4]	4.41	1350
I	3	58	39.0	35.8	4.65	2380
J	3	56	45.8	44.2	4.31	900
C	4	62	46.5	43.2	4.60	2050
D	4	59	58.8	46.2	[4]	7180
E	4	59	48.7	45.1	4.10	2070
F	4	59	48.5	42.3	4.60	2580
G	4	55	36.8	36.7	3.92	760
H	4	59	[4]	[4]	4.41	1700
I	4	59	41.7	36.5	4.65	2850
J	4	55	49.5	48.2	4.31	580
K	[3]	60	49.0	46.0	5.85	704

[1] Code identification of trade name and supplier in Table 9

[2] Plasticizer identification 1 = 10 phr dibutyl phthalate; 2 = 6.5 phr FLEXOL 4-GO, 3.5 phr Santicizer 140; 3 = 4.5 phr FLEXOL 4-GO, 10.5 phr Santicizer 140; 4 = 20 phr Santicizer 140

[3] Identity of plasticizer not disclosed by manufacturer

[4] Data not available

TABLE 12

FILTERABLE SOLIDS CONTENT OF VARIOUS COMMERCIAL POLYVINYL ACETATE LATEXES

Latex[1]	Filterable Solids, Percent			
	As Is	Formulation A[2]	Formulation B[2]	Formulation C[2]
A	0.0099	--[3]	--	--
B	0.0058	--	--	--
C	0.0031	0.0043	0.0050	0.0118
D	0.0101	0.0164	0.0145	0.0253
E	0.0021	0.0228	0.0045	0.0075
F	0.0110	0.0159	0.0219	0.0389
G	0.0005	0.0034	0.0049	0.0071
H	0.0092	0.0208	0.0217	0.0155
I	0.0063	0.0073	0.0076	0.0128
J	0.0002	0.0003	--	0.0086
K	0.0123	--	--	--

[1] Code identification of commercial latexes in Table 9

[2] Formulation A: 6.5 phr FLEXOL 4-GO, 3.5 phr Santicizer 140
Formulation B: 10.5 phr FLEXOL 4-GO, 4.5 phr Santicizer 140
Formulation C: 20 phr Santicizer 140

[3] Dashes indicate test is not applicable or data are not available

TABLE 13

STORAGE STABILITY AT 140° F

Formulation	Tensile Strength, psi		Elongation, %	
	Control	140° F Storage	Control	140° F Storage
3.5 phr Santicizer 140 6.5 phr FLEXOL 4-GO	1760	1675	260	230
4.5 phr FLEXOL 4-GO 10.5 phr Santicizer 140	1535	1470	270	270
20 phr Santicizer 140	1200	1190	280	310

TABLE 14

SHEAR STABILITY OF COMMERCIAL POLYVINYL ACETATE LATEXES AFTER PLASTICIZATION

Latex [1]	Plasticizer [2] Formulation	Filterable Solids, Percent		Viscosity, cps	
		Before Shear	After Shear	Before Shear	After Shear
A	D	0.0490	0.080	2300	2460
B	[3]	0.0058	0.0249	2900	3060
C	A	0.0043	0.0029	2400	3600
C	B	0.0050	0.0074	2420	3630
C	C	0.0118	0.0018	2050	2500
D	A	0.0164	0.0180	6300	[5]
D	B	0.0145	0.0191	7650	[5]
D	C	0.0253	0.0085	7180	[5]
E	A	0.0228	0.0336	2100	2000
E	B	0.0046	0.0111	2185	2050
E	C	0.0075	0.0050	2270	2080
F	A	0.0159	0.0096	2660	2380
F	B	0.0210	0.0140	2524	2268
F	C	0.0380	0.0069	2580	2380
G	A	0.0035	0.0270	1380	[4]
G	B	0.0049	0.0160	1035	[4]
G	C	0.0072	0.0180	760	[4]
H	A	0.0200	0.0401	1010	1190
H	B	0.0217	0.0274	1350	1410
H	C	0.0155	0.0051	1700	2100
I	A	0.0074	0.0184	1880	2010
I	B	0.0077	0.0159	2380	2490
I	C	0.0128	0.0168	2848	3100
J	A	0.0002	0.0120	1200	1180
J	B	0.0138	0.0148	900	920
J	C	0.0086	0.0098	580	600

[1] Code identification of commercial latex in Table 9

[2] Formulation A: 6.5 phr FLEXOL 4-GO, 3.5 phr Santicizer 140
Formulation B: 10.5 phr FLEXOL 4-GO, 3.5 phr Santicizer 140
Formulation C: 20 phr Santicizer 140
Formulation D: 10 phr Dibutyl phthalate

[3] Plasticizer not revealed by manufacturer

[4] Too much foam to allow viscosity measurement

[5] Too difficult to handle to obtain an accurate viscosity reading

TABLE 15

SUMMARIZED RECOMMENDED SPECIFICATIONS

Property	Test Method	Specification
Composition	--	Polyvinyl acetate latex with 6.5 phr FLEXOL 4-GO and 3.5 phr Santicizer 140
Tensile Strength	ASTM D 822 (1)	1326 psi, minimum (2)
Elongation	ASTM D 822 (1)	226%, minimum (2)
Total Solids	ASTM D 1417-67 (1)	55 to 57%
Surface Tension	ASTM D 1417-67 (1)	40.0 dynes/cm. minimum (2:1 dilution)
pH	ASTM D 1417-67 (1)	4.0 to 6.0
Viscosity	ASTM D 1417-67 (1)	800 to 3000 cps.
Filterable Solids	See page 23	0.03% maximum
Salt Tolerance	See Page 25	0.1% maximum
Storage Stability at 140°F	See Page 25	Must retain 80% of initial tensile strength and elongation
Shear Stability	See page 25	Viscosity change, 50% maximum. Filterable solids, 0.03% maximum after treatment. Foam volume, 20% maximum after treatment.

(1) Note modifications described in text.

(2) In addition, any product from new supplier must undergo qualification procedure.

APPENDIX A:

WES LABORATORY PROCEDURES AND TESTS FOR SCREENING OF PROPOSED DUST-CONTROL MATERIALS

1. Soil types. The following three soils will be used in the laboratory evaluation tests of dust-control materials:

   a. Fine sand (SP) .
   b. Silt (CL) .
   c. Heavy clay (CH) .

2. Test specimens. From the above test soils, soil specimens will be prepared at various initial conditions as follows and used to evaluate the effectiveness of a proposed dust palliative:

   a. Specimen A - Sand in a loose, dry state.
   b. Specimen B - Silt in a loose, dry state.
   c. Specimen C - Heavy clay in a loose, dry state.
   d. Specimen D - Silt, compacted and dried back.
   e. Specimen E - Heavy clay, compacted and dried back.

3. Processing of soils for molding. The fine sand will be dried to a water content of 1 percent or less. To prepare the silt and heavy clay for the loose, dry specimens, each soil will be processed to pass the No. 40 sieve, the material passing to be dried to water contents of 3 percent or less and 9 percent or less, respectively. Processed in this manner, a large fraction of each test soil will be dust. To prepare the silt and heavy clay for the compacted specimens, each soil will be processed through a No. 4 sieve, the material passing to be brought to a water content of 18 and 28 percent, respectively. The latter two soils will be allowed to equilibrate at least 24 hours prior to molding.

4. Molding procedures. Molds, 6 in. square by 3 in. deep, will be used to contain the soil specimens. The soil for specimens that are to be prepared in a loose, dry state will be placed in a mold without any compaction effort. The remaining silt and heavy clay specimens will be prepared by statically compacting a preweighed quantity of the processed soil to achieve a dry density of 90 lb per cu ft, then surface dried for 1 hour under a heat lamp or similar means at a surface temperature of 120°F.

5. Prewetting of specimen surfaces. For most surface-applied materials, the surfaces of the specimens may require prewetting prior to treatment to break existing surface tensions. A simple test will be conducted consisting of placing the loose, dry soils in paper cups and applying a quantity of the dust-control material. If it is apparent that the material does not achieve complete coverage and is not absorbed by the soil surface, prewetting with water will be necessary. Prewetting of the prepared soil specimens will be accomplished by fog spraying with water at a rate sufficient to slightly dampen the surface of the specimen. In general a rate of 0.03 to 0.10 gal per sq yd will provide the necessary prewetting.

6. Application procedures. Application will be accomplished with a specially devised laboratory spray device whenever the material is capable of being dispensed in this manner. If the viscosity of the palliative prohibits this spray device, other techniques will be designed.

7. Treatment level. Materials will be applied and examined initially at a rate of treatment and dilution as specified by the supplier, but not to exceed 5 lb of material as-supplied (i.e., exclusive of diluent) and not to exceed 2 gal per sq yd of diluted material. If the supplier's recommended rate of application is less than 5 lb per sq yd and failure occurs under the subsequent tests, the level of treatment will be increased to 5 lb per sq yd of material as-supplied. Whenever the rate of treatment and dilution is not specified by the supplier, an application rate of 5 lb per sq yd of material as-supplied will be used. All samples that pass the complete test cycle at a rate of application of 5 lb per sq yd will be retested at an application rate of 3 lb per sq yd as-supplied.

8. Cure. All treated specimens will be cured in the laboratory for 4 hours under intermediate climatic conditions of 77°F (± 5°F) and 50 percent (± 10 percent) relative humidity. In the event the material has not cured after 4 hours, it will be cured for 24 hours prior to testing.

9. Impingement test. At the end of the cure period, the treated specimens will be subjected, successively, to air blasts generating stagnation pressures of 7, 30, and 55 lb per sq ft. Each air blast will be sustained for a duration of 1 minute and directed to impinge the treated surface at an angle of 20 degrees.

10. Rainfall erosion test. All treated specimens surviving the air-impingement test will be subjected to a simulated rainfall erosion test for a period of 1 hour. All specimens surviving this test will be resubjected to the air-impingement test immediately following the rainfall erosion test.

11. Drying cycle. Specimens surviving all preceding tests will be air-dried at ambient laboratory conditions for an additional 16-20 hours, then placed under a heating device for 1 hour at a surface temperature of 120°F. The specimens will then be resubjected to the air-impingement test.

12. Data obtained. Data obtained from the laboratory test will include:

a. Water content and dry density of the prepared soil specimen.

b. Depth of penetration versus time during the curing period.

c. Ability to withstand air impingement at the three levels of wind velocity.

d. Ability to withstand the water-erosion test.

e. Ability to withstand air impingement at the three levels of wind velocity following the water-erosion test.

f. Supplementary observations pertinent to the ease of working with and applying the material, the condition of the surface after curing and testing, flexibility of the surface, and other comments which may be significant to the overall evaluation of the material.

13. Second-phase testing. All specimens surviving the aforementioned tests will be considered for further testing and evaluation under the helicopter downwash facility and/or field tests under traffic.