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The Effect of Compaction on the Storage Stability of DDT Water-Dispersible Powders *

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Tests have been carried out to determine whether DDT powder might be subjected to greater compaction than is now the rule without affecting the quality of the product, particularly as regards its storage stability. Such increased compaction would make it possible to pack the powder in smaller containers for any given quantity and thus to achieve considerable savings in shipping and handling costs.

The tests described in this paper showed that, while suspensibility of the powder is little affected by pressures of up to 45 p.s.i., considerable deterioration of suspensibility occurs when compacted samples are heated to 50°C—temperature encountered in conditions of tropical storage.

The authors conclude that DDT powders currently produced would be seriously damaged if stored for long periods at high temperatures while under pressure. For this reason they recommend that bags should not be used for shipment of DDT water-dispersible powders in the place of the polyethylene-lined fibre drums at present in general use.

In current practice, technical DDT is mixed with wetting and dispersing agents and ground with an inert diluent in an air mill to produce a very fine, highly suspensible powder which is readily dispersed in water, Powders of this type manufactured for use in malarious areas of the world must have excellent suspensibility characteristics because they are applied with hand-operated spraying equipment. In addition, the powders must be able to withstand shipment over long distances and storage for long periods under tropical conditions. In order to afford as much protection to the powder as possible, DDT water-dispersible powders purchased by the International Cooperation Administration are required to be packaged in self-supporting, polyethylene-lined fibre drums with a minimum capacity of 26 US gallons for 100 pounds of powder.[4] Immediately after air milling, the bulk density of DDT powder is very low and some compaction is usually necessary in order to pack 100 pounds in the 26-gallon container. This is accomplished by application of vacuum or by other mechanical means. Further compaction could be accomplished by the formulator; but this has not been recommended, since little is known about the effect of compaction on the storage life of the powder except that extreme compaction seriously affects the suspensibility.

The purposes of this investigation were to determine the effect of compaction on the storage stability of DDT water-dispersible powders, and to determine whether a smaller container or another type of container could be used for shipment of the powders without affecting the quality. Considerable savings in shipping costs could be effected if the volume of the 100-pound drum could be reduced below 26 gallons. Even more savings would result if the powder could be shipped in bags; but if bags were used, the powder in the bottom bag of a stack would be subjected to the pressure of the powder in the bags stacked upon it during shipment and storage.

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[4] 1 US gallon = 3.79 litres; 1 pound = 0.45 kg.

LABORATORY PRESSURE TESTS

The first experiments with pressure were conducted on a laboratory scale. A special mortar was constructed similar to a diamond mortar, with a base and piston made of brass and a sleeve of stainless steel. The diameter of the piston was 1 inch (2.54 cm) and the mortar depth 1½ inches (3.81 cm). Ten-gram samples of the powders were used. A sample was placed in the mortar, the piston was inserted, and weights were placed on top of the piston to produce the desired pressure and a resultant compaction. Pressures (in excess of atmosphere) of 15, 30, 45, and 75 pounds per square inch (p.s.i.) [1] were tested. A given weight was applied to a sample for a one-minute period. The height of the piston in the mortar was then measured and the volume of the compacted powders was calculated. Next, the sample was removed from the mortar, weighed, and the bulk density calculated. Each sample was crushed, with a minimum of grinding action, and tested for suspensibility (expressed as a number representing the weight to volume percentage of DDT in suspension) by the procedure outlined in ICA Specification 72059.[2]

Data from these tests are presented in Table 1. Four different formulations were available for testing. Data from the first three formulations represent average suspensibility values from five batch samples. Data from the fourth formulation are based on a single batch sample. From these data, it appears that pressures of the order of 15-45 p.s.i. have little effect on the suspensibilities of DDT water-dispersible powders. In fact, in most cases the suspensibilities were found to be higher after application of pressure. The volumes which would be occupied by 100 pounds of powder were calculated from the bulk densities after compaction, and these values are presented in Table 2.

To determine the effect of heat on the compacted powders, the above experiment was repeated and the compacted samples were stored in a 50°C oven for three weeks. At the end of this period, the powders were removed and their suspensibilities determined. A non-compacted sample carried through the same procedure served as a control.

Data from the experiment are presented in Table 3. Values from five batch samples were averaged to give the data presented for formulations 1, 2, and 3, while the values for formulation 4 are based on a single sample.

TABLE 1

SUSPENSIBILITY AFTER COMPACTION AT PRESSURES INDICATED

Formulation	As received	15 p.s.i.	30 p.s.i.	45 p.s.i.	75 p.s.i.
1	1.38	1.50	1.49	1.62	1.55
2	1.26	1.40	1.44	1.61	1.34
3	1.93	2.03	1.96	2.09	1.86
4	1.98	2.09	1.90	2.12	1.65

TABLE 2

VOLUMES (IN US GALLONS) OCCUPIED BY 100 POUNDS OF POWDER AFTER COMPACTION

Formulation	15 p.s.i.	30 p.s.i.	45 p.s.i.	75 p.s.i.
1	20.7	19.4	18.5	17.5
2	19.7	18.5	17.7	16.8
3	20.7	19.1	18.6	17.1
4	20.1	18.9	18.3	17.0

TABLE 3

EFFECT OF HEAT ON COMPACTED SAMPLES

Formulation	Suspensibility			
	No pressure	15 p.s.i.	45 p.s.i.	75 p.s.i.
1	1.53	1.32	1.27	1.04
2	1.35	1.39	1.10	0.92
3	1.96	1.93	1.62	1.21
4	—	1.62	1.43	1.09

From these experiments, it may be concluded that while pressures of 15-45 p.s.i. have little or no immediate effect on suspensibility, samples so compacted are subject to serious deterioration when heated, even though the pressure is removed during the heating period. Since suspensibility is a function of particle size, it appears that compaction of the powder tends to promote fusion of the particles. This is an example of irreversible agglomeration described by Miles et al.[3] which results in lowered suspensibility values.

TESTS ON POWDERS PACKED IN BAGS

In order to determine whether bags could be used for shipment of DDT water-dispersible powders,

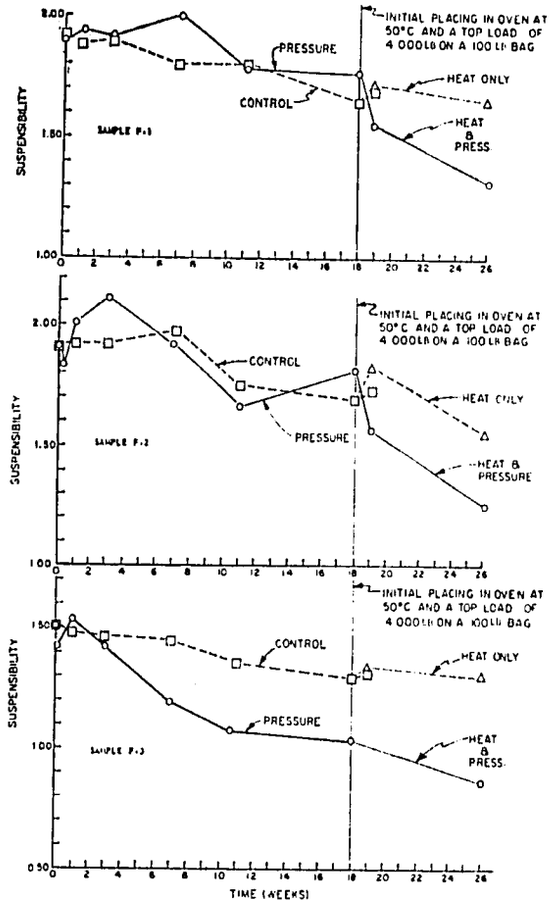
[1] 1 p.s.i. = 0.07 kg/cm².

[2] International Cooperation Administration (1959) *DDT, 75 percent, water-dispersible powder*, Washington, D.C. (ICA Specification 72059, 20 July 1959).

[3] Miles, J. W., Goette, M. B., Grimm, L. & Pearce, G. W. (1959) *Bull. Wld Hlth Org.*, **20**, 937.

FIG. 1

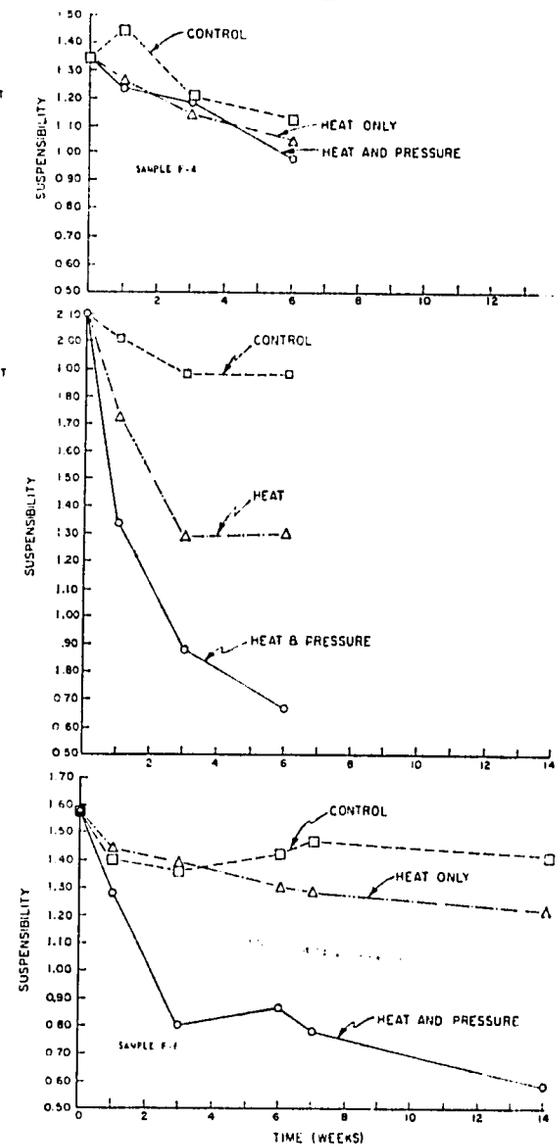
EFFECT OF PRESSURE AND EFFECT OF HEAT PLUS PRESSURE ON SUSPENSIBILITY OF DDT WATER-DISPERSIBLE POWDER



three 100-pound drums containing fresh production samples were obtained from different formulators and repacked in specially prepared polyethylene-lined burlap bags. A one-pound sample from each 100 pounds was packed in a sealed glass jar to serve as a control. The bags were placed in a specially constructed press and subjected to a top load of 4000 pounds, equivalent to the pressure exerted on the bottom bag in a stack of 100-pound bags piled 40 bags high. This was considered to be the maximum height to which the bags would be likely to be stacked. Pressure on the bags was maintained with a mechanical screw-type jack and compression spring and measured with an electronic strain gauge. Since the average area of a bag lying flat is 560 square inches (3613 cm²), a static pressure of 4000 pounds is equivalent to about 7 p.s.i. The tests were conducted at ambient temperature in a non-air-conditioned laboratory. Samples were taken over a period of 18 weeks and their suspensibility values compared with the control samples. Data from these tests are presented graphically in Fig. 1.

FIG. 2

EFFECT OF HEAT AND EFFECT OF HEAT PLUS PRESSURE ON SUSPENSIBILITY OF DDT WATER-DISPERSIBLE POWDER



At the conclusion of these tests, the entire press assembly and the test samples were transferred to a large temperature-controlled cabinet maintained at 50°C. The 4000-pound static pressure was maintained throughout this test. Changes in suspensibility after application of heat are also presented graphically in Fig. 1. The tests were continued for an additional eight weeks in the heated cabinet.

Conclusions drawn from these tests parallel those from the laboratory-scale tests. Samples suffer little or no damage from the application of moderate pressures; however, a serious reduction in suspensibility occurs when the compacted samples are heated. Sample F-3 in Fig. 1 did show more decrease in suspensibility by pressure alone than did the control; however, this particular sample was not considered to be representative of powders purchased by ICA in that the initial suspensibility was below the minimum required by the current specification.

In a second series of tests 100-pound samples of DDT 75% water-dispersible powder from three formulators were packed in polyethylene-lined burlap bags and placed in the special press under a top load of 4000 pounds. The whole assembly was then placed in a cabinet maintained at 50°C. One pound of each sample held in a sealed glass jar at ambient temperature served as a control, and a second one-pound sample was placed in the 50°C cabinet to determine the effect of heat on the material. Samples were removed after 1, 3, 6, 7, and 14 weeks and their suspensibility determined. Data from these tests are presented in Fig. 2.

CONCLUSION

From the data from both series of tests it is concluded that DDT powders currently in production would be seriously damaged if stored for long periods of time at high temperatures while under pressure. For this reason, it was recommended that bags should not be used for shipment of DDT water-dispersible powders.

RÉSUMÉ

En raison de l'extrême finesse qui assure leur excellent pouvoir de dispersion dans l'eau, les poudres de DDT occupent un volume considérable sitôt après broyage. Leur densité augmente peu à peu lorsqu'elles sont laissées au repos. Il est possible d'accélérer ce processus par vibration mécanique ou traitement par le vide. Les fabricants réduisent habituellement le volume de la poudre par un moyen mécanique quelconque, afin que le produit puisse être emballé selon les exigences des usagers, dans des tambours recouverts intérieurement de polyéthylène, contenant 45 kg dans environ 100 l (100 lb/26 US gallons). On pourrait diminuer les frais de transport en augmentant la densité de la poudre, mais l'on ne dispose guère d'informations relatives à l'effet de la réduction de volume sur la stabilité de la poudre au cours du stockage.

Les auteurs ont cherché à préciser ces effets, et ont mis à l'étude l'emploi d'autres emballages — tambours plus petits, ou sacs. Au cours d'essais en laboratoire, à échelle réduite, ils ont soumis des échantillons de poudre de DDT dispersable dans l'eau à des pressions allant jusqu'à 5,5 kg/cm². On a constaté que des pressions allant jusqu'à 3,15 kg/cm² n'ont pas d'effet ou un effet négligeable, mais que le même traitement provoque une baisse sérieuse de l'aptitude à se mettre en suspension, si la poudre est conservée pendant longtemps à 50° C.

Dans une autre série de tests, des sacs doublés de polyéthylène contenant 45 kg (100 lb) de poudre à 75% de DDT dispersable dans l'eau ont été soumis à une pression totale de 1800 kg. Cette pression est obtenue en disposant 40 sacs de 45 kg sur le sac test, ce qui donne une pression de 0,5 kg/cm² sur la poudre. La poudre utilisée pour cette expérience provient de la production courante fournie à l'International Cooperation Administration de Washington: son aptitude à se mettre en suspension n'est pas modifiée après avoir été maintenue sous cette pression à température ambiante, alors qu'elle est nettement diminuée si cette même pression est exercée à 50° C.

Ces expériences permettent de conclure que la poudre de DDT dispersable dans l'eau, de fabrication courante, serait sérieusement endommagée par un magasinage prolongé à haute température et sous pression, et qu'il est recommandé de ne pas utiliser de sacs pour le transport d'une telle poudre.

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