DRUG DEVELOPMENT AND INDUSTRIAL PHARMACY, 10(3), 505-514 (1984)

A SIMPLE METHOD FOR DIFFERENTIATING SOURCES OF PREGELATINIZED STARCH NF

Frank E. Bowen and Winston A. Vadino

Pharmaceutical Research Division,
Schering-Plough Corporation, Bloomfield, NJ 07003

ABSTRACT

A simple test procedure to differentiate between sources of pregelatinized starch NF has been developed. The procedure is based on the swelling characteristics of the materials. Three pregelatinized starch NF products were distinguished by observing their sedimentation volumes. Microscopic differences in the particle morphology of each starch product were also observed. The study indicates that sedimentation testing can be used as an addition to the current compendial identity tests for pregelatinized starch NF.

INTRODUCTION

Formulation advantages for the use of modified or pregelatinized corn starch have been clearly recognized by the pharmaceutical industry (1-7). The compendial monograph (8) for pregelatinized starch describes the material as starch which has been chemically or

0363-9045/84/1003-0505$3.50/0

mechanically processed to cause partial or complete hydrolysis of the starch grains. The degree of hydrolysis depends on the manufacturer's processing technique. Although excipients from alternate sources may be chemically equivalent by compendial standards, there is cause to note that they may exhibit different physical characteristics (9-13).

The current compendial tests fail to differentiate sources of pregelatinized starch NF. A test for determining the percentage of cold water solubles, requiring approximately four hours, has been used to identify one product (14).

A comparison of sedimentation volumes has been used to macroscopically compare the swelling capacity of several disintegrating agents (10,15). Appearing to be a simple procedure, the use of sedimentation testing was investigated as a means of identifying different sources of pregelatinized starch NF.

MATERIALS

Samples of three pregelatinized starch NF products were obtained. They were Starch 1500™ (formerly Sta-Rx 1500, Colorcon Inc., West Point, PA), National 1551 and National 78-1973 (National Starch and Chemical Co., Bridgewater, NJ).

METHOD

Sedimentation Test

One gram of pregelatinized starch NF was dispersed in 45 ml of distilled water (20° ± 5°C) using a 100 ml glass beaker, magnetic stirring bar and magnetic stirring plate. After 5 minutes of mixing, the dispersion was poured into a 50 ml graduated cylinder (PYREX,

Corning No. 3046). An additional 5 ml of distilled water was used to rinse the beaker, and the rinse then added to the cylinder. The cylinder was allowed to stand undisturbed. Sedimentation volumes were observed after 30, 60, 90 minutes and 24 hours.

Effect of Mixing Time

Sedimentation testing was performed on dispersions mixed for 30 seconds, 5 and 30 minutes.

Effect of Water Temperature

Sedimentation testing was performed with dispersions prepared using water at both 5°C ± 3° and 45°C ± 5°.

Effect of Product Aging

Samples of each product were stored for two weeks at 55°C, 75°C and 35°C/75% relative humidity prior to sedimentation testing.

Photomicrographs

Photographs of each product suspended in water were taken at 130X magnification to observe product morphology.

RESULTS AND DISCUSSION

Sedimentation testing was performed using three lots of each pregelatinized starch product. As can be seen by the data presented in Table 1, the observed sedimentation volumes of National 1551 were approximately three times greater than National 78-1973 and approximately double that of Starch 1500™. Lot-to-lot variation was minimal for each product. It is obvious from this simple procedure that chem-

TABLE 1

Effect of Standing Time on Sedimentation Volume

	Sedimentation Volume, ml[a]			
	30 min	60 min	90 min	24 hr
Starch 1500™ Lot. 202020	8	8	8	8
National 1551 Lot. CB 7041	14	14	15	15
National 78-1973 Lot. 4370-35	5	5	5	5

[a] Recorded to the nearest milliliter.

TABLE 2

Effect of Mixing Time on Sedimentation Volume

		Sedimentation Volume, ml[a]		
	Lot No.	30 sec	5 min	30 min
Starch 1500™	202020	8,8	8,8	8,9
	202023	8,8	8,8	9,9
	205011	9,9	9,9	9,9
National 1551	CB7041	15,15	15,15	15,15
	GA7314	15,15	15,15	15,15
	HA7348	13,13	13,13	14,14
National 78-1973	4370-35	5,5	5,5	5,5
	4370-42	5,5	5,5	5,5
	4370-55	5,5	5,5	5,5

[a] Duplicate tests; recorded to the nearest milliliter.

TABLE 3

Effect of Water Temperature on Sedimentation Volume

	Lot No.	Sedimentation Volume, ml[a] 20°C±5°	5°C±3°	45°C±5°
Starch 1500™	202020	8,8	7,7	10,10
	202023	8,8	7,7	10,11
	205011	9,9	8,8	10,11
National 1551	CB7041	15,15	13,13	16,16
	GA7314	15,15	13,14	16,16
	HA7348	13,13	12,12	15,15
National 78-1983	4370-35	5,5	5,5	5,5
	4370-42	5,5	5,5	5,5
	4370-55	5,5	5,5	5,5

[a] Duplicate tests; recorded to the nearest milliliter.

ically equivalent products can be differentiated by inherent physical behavior.

Data in Tables 1-3 were developed to establish test procedure parameters. The data in Table 1 show that a standing time of 90 minutes was necessary to attain a constant sedimentation volume.

Apparently due to the almost instantaneous swelling of the starch grains (10,16), sedimentation volumes were not affected by the mixing time of the starch dispersion (Table 2). Dispersions agitated for 30 seconds or 30 minutes attained similar heights.

Data showing the effect of water temperature on sedimentation volume are presented in Table 3. Some minor volume differences in "cold"

TABLE 4

Effect of Two Weeks Storage of Pregelatinized Starch at Accelerated Conditions on Sedimentation Volume

		Sedimentation Volume, ml[a]		
	Lot No.	55°C	75°C	35°C/75%RH
Starch 1500™	202020	8,8	8,8	8,8
	202023	9,9	8,9	8,9
	205011	9,9	9,9	9,9
National 1551	CB7041	14,14	14,14	14,14
	GA7314	14,14	14,14	14,15
	HA7348	13,13	13,13	13,13
National 78-1973	4370-35	5,5	5,5	5,5
	4370-42	5,5	6,6	5,6
	4370-55	5,5	5,5	5,5

[a] Duplicate tests; recorded to the nearest milliliter.

(5° ± 3°C) and "warm" (45° ± 5°C) water were noted with Starch 1500™ and National 1551 but no changes were observed to National 78-1973 samples. The height variations occurring with samples containing a greater percentage of hydrolyzed starch may be attributed simply to the thermal expansion and contraction of water. The temperatures studied were sufficiently low as to not cause any significant swelling of unhydrolyzed starch (17).

Pregelatinized starch samples stored at elevated temperature and temperature/humidity conditions were also evaluated. The observed data are presented in Table 4. As can be seen, sedimentation volumes of the three starches were apparently unaffected by the accelerated storage conditions used in this study.

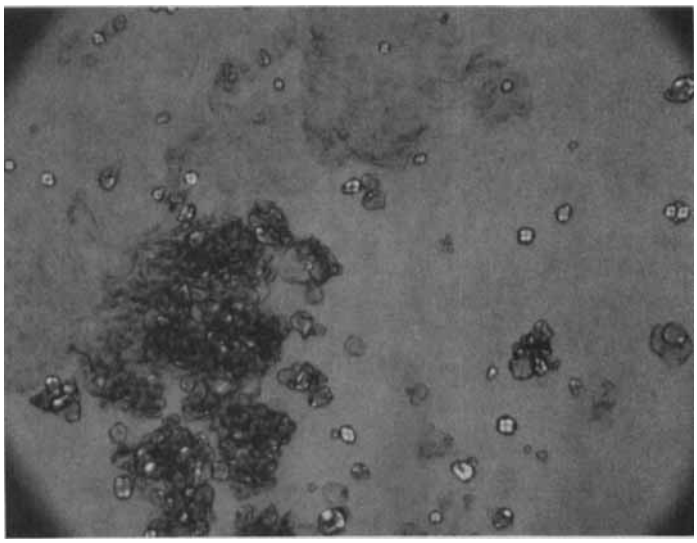

FIGURE 1

Starch 1500™

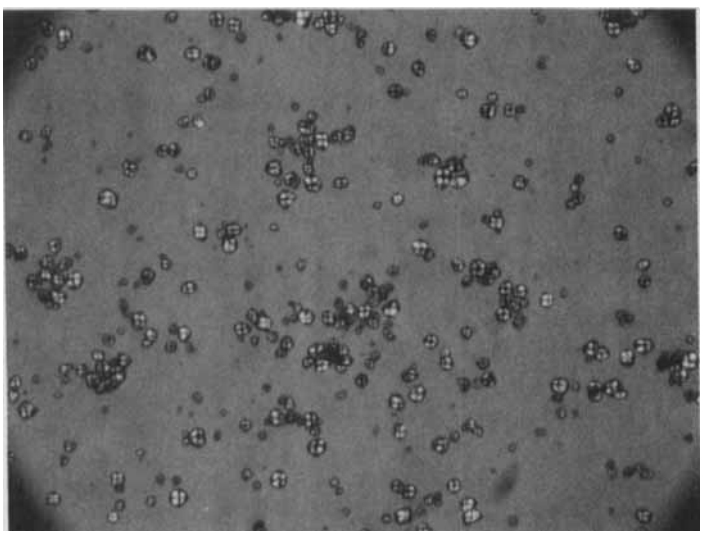

FIGURE 2

National 78-1973

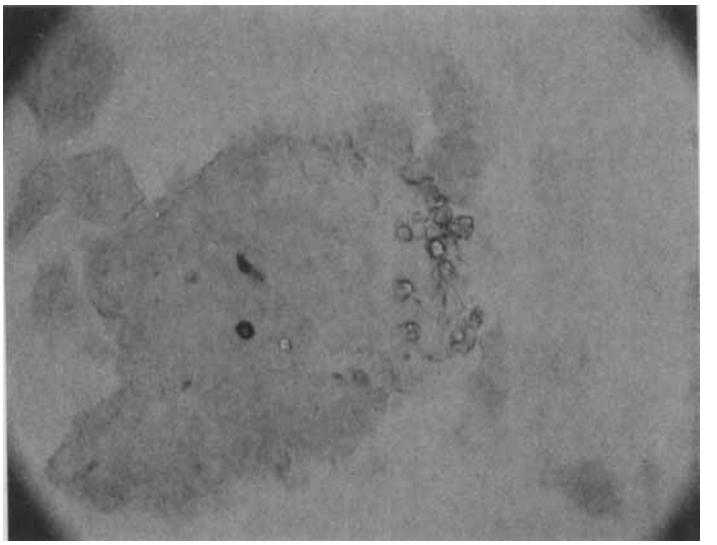

FIGURE 3
National 1551

The photographs in Figures 1-3 indicate the unique morphological characteristics created by the individual manufacturing processes. Both Starch 1500™ (Figure 1) and National 78-1973 (Figure 2) consist of a mixture of individual starch grains and grain aggregates bound to hydrolyzed starch. National 78-1973 exhibited a minimum degree of gelatinization while National 1551 (Figure 3) appeared to be almost completely gelatinized.

CONCLUSION

Using sedimentation volumes, a simple method has been established for differentiating sources of pregelatinized starch NF. The method was validated by testing several lots of each pregelatinized starch product. The effect of several procedural parameters on sedimentation volume was also evaluated. Test results were not influenced by accel-

erated environmental storage conditions of the pregelatinized starch. The procedure can be used as a quality control addition to current compendial tests.

The differences noted between the three pregelatinized starch NF products emphasize the caution formulators and production areas should consider when planning to use comparable materials from alternate suppliers. The observed differences in physical properties of each pregelatinized starch may represent functional variables which could only be evaluated in an actual formulation or manufacturing process.

ACKNOWLEDGEMENTS

The authors would like to thank Mr. John Teague for preparing the photomicrographs.

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