ASCORBIC ACID (VITAMIN C) adds nutritive value to wheat flour, corn-soya-milk (CSM), and precooked infant cereals. Addition of ascorbic acid may be desirable when these foods are intended for use in diets normally deficient in this vitamin. Specifications for the purchase of CSM by USDA have now been revised (1) to include the addition of 364 g. of ascorbic acid per ton of final food blend (182 mg. per lb.).

Since consideration is being given to whether ascorbic acid should also be added to wheat flour for export and to infant cereals for domestic distribution, experiments were carried out in which wheat flour, CSM, and precooked infant cereals were fortified with ascorbic acid and stored at different moisture levels and temperatures. Ascorbic acid retention was correlated with these variables.

Materials
From a blend of Kansas HRW wheats, milled on a Miag Multomat pneumatic experimental flour mill, a patent flour of about 60% extraction (wheat basis) was prepared. Flour at 14.6% moisture was produced by tempering wheat to 20% moisture before milling. Flour at 13.7% moisture was obtained by tempering wheat to 17% moisture before milling, and flour at 12.9% moisture by air-drying the flour. Each flour was equilibrated for at least 7 days and then fortified with 400 mg. of ascorbic acid per lb. of flour. Uncoated ascorbic acid of USP-FCC grade (brand A) and 97.5% ascorbic acid coated with 1% ethyl cellulose from the same manufacturer were tested and compared. Uncoated ascorbic acid USP-FCC grade (brand B) from another source was tested and compared with brand A and also used in storage tests with infant cereals.

CSM used in the tests was a commercial product containing 9.2% moisture and 2.0% fat made up with 68% processed low-fat corn meal, 25% extracted and toasted soy flour, 5% nonfat dry milk, and 2% vitamins and minerals. CSM at 8.0% moisture was prepared by air-drying this commercial CSM. CSM blends at 10.4 and 11.8% moisture were prepared by atomizing water and spraying it on the CSM spread in thin layers. The blends were equilibrated for several days and then fortified with 200 mg. of either brand A (uncoated) or coated ascorbic acid per lb. of CSM.

In one experiment precooked infant cereal was prepared by heating a slurry containing oat, corn, and wheat flours. A small amount of barley malt was added after the temperature of the slurry reached 155°F. The mixture was first held for 3 min. at this temperature, then heated to boiling, and boiled for 15 min. Next, a slurry containing extracted and toasted soy flour, sucrose, vitamins, and minerals (including brand B ascorbic acid) was mixed into the hot cooked cereals, and this mixture was drum-dried on steam-heated rolls with steam at 40 p.s.i.g. The dried material was coarsely ground on corrugated rolls. The final product contained 40% oat flour, 18.5% corn flour, 18.5% wheat flour, 14% soy flour, 6% sucrose, and 3% vitamins and minerals, and had an ascorbic acid content of 600 to 800 mg. per lb. One portion of the infant cereal was tested as produced, at 7.0% moisture; another was dried to 5.0% moisture; and a third portion was exposed to a humid atmosphere to raise its moisture content to 10.7% before overnight equilibration.

C. Vojnovich and V. F. Pfeifer
Northern Regional Research Laboratory
Peoria, Illinois 61604

Stability of Ascorbic Acid in Blends with Wheat Flour, CSM, and Infant Cereals

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Contribution from Northern Regional Research Laboratory, Peoria, Ill. 61604. This is a laboratory of the Northern Utilization Research and Development Division, Agricultural Research Service, U.S. Department of Agriculture. Mention of trade products is for identification only and does not imply endorsement by the Department.
In a second experiment the infant cereal was prepared as described, but ascorbic acid was omitted from the cook. The finished cereal flakes were adjusted to moisture contents of 4.8, 6.5, and 10.8% as described, and dry-mixed with brand B acid to a level of 600 mg. per lb.

Particle size analyses of the ascorbic acid products used are given below:

<table>
<thead>
<tr>
<th>U.S. Standard Sieve, Mesh</th>
<th>Uncoated Ascorbic Acid USP-FCC Grade Brand A</th>
<th>Ascorbic Acid Coated with 1% Ethyl Cellulose Brand B</th>
</tr>
</thead>
<tbody>
<tr>
<td>On 20</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>40</td>
<td>0</td>
<td>26</td>
</tr>
<tr>
<td>60</td>
<td>22</td>
<td>37</td>
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<tr>
<td>80</td>
<td>19</td>
<td>22</td>
</tr>
<tr>
<td>100</td>
<td>12</td>
<td>10</td>
</tr>
<tr>
<td>Thru 100</td>
<td>47</td>
<td>5</td>
</tr>
</tbody>
</table>

Methods and Analyses

Samples of the various cereal blends were placed in 8-oz. glass bottles, an air space was left in each bottle of about one-fourth the volume, and the bottles were sealed with paraffin wax to prevent loss of moisture. Storage temperatures for all blends were 26°, 37°, and 45°C. Wheat-flour samples were stored at moisture contents of 12.9, 13.7, and 14.6%; CSM samples at 8.0, 10.4, and 11.8%; and samples of precooked infant cereal at about 5.0, 7.0, and 10.7%. Individual bottles were removed at intervals and their contents analyzed for ascorbic acid.

Ascorbic acid in each sample was determined by the indophenol-xylene extraction method, AACC Method 86-10 (2). Moisture was determined by the Brabender oven method at a temperature of 120°F. for 45 min.

Results and Discussion

Since data from all storage tests can be represented well by straight lines on semilog plots, the kinetics covering the rate of destruction of ascorbic acid in all cereal blends are similar to those of a unimolecular or first-order reaction. Destruction rates were calculated according to the formula

\[ k = \frac{2.3}{t} \log \frac{C_0}{C} \]

where \( k \) is the rate of destruction of ascorbic acid at the given temperature, \( C_0 \) is the acid concentration at the beginning of storage at that temperature, \( C \) is the acid concentration after \( t \) in weeks. The rates are listed in Table I. When these rates were plotted against the reciprocals of the absolute temperatures in an Arrhenius-type plot, straight lines were obtained from which the rate of destruction at any temperature could be estimated.

Results with Wheat Flour

The corresponding ascorbic acid destruction rates in Table I. Storage data show that at 45°C, the rate of destruction of ascorbic acid increases greatly as moisture content increases from 12.9 to 14.6%. At 37°C the acid was moderately stable at all three moisture levels and, at 26°C., was extremely stable at all three moisture levels. The studies summarized in Fig. 1 show clearly that brand A was

![Fig. 1. Storage of wheat flour containing uncoated USP-FCC-grade ascorbic acid (brand A); effect of moisture content and temperature on retention of ascorbic acid.](image_url)

![Table I. Destruction Rates for Ascorbic Acid Stored in Wheat Flour, CSM, or Mixed Cereal](table_image)

\[ a_k = (2.3/t) \log (C_0/C) \] (see text).
quite stable in wheat flour when the moisture content was about 13% or less. At higher moisture levels this USP ascorbic acid was unstable at 45°C. From an Arrhenius-type plot, and a subsequent plot of destruction rate vs. moisture content, it was indicated that wheat flour at 13.7% moisture had good stability when stored at 40°C—70% retention after 18 weeks of storage.

Destruction rates from tests in which flour at 13.7% moisture was fortified with coated 97.5% acid are also listed in Table I. The data show that the coated acid had much better storage stability at 45°C, and somewhat better stability at 37°C and 26°C than brand A. Since particle-size distributions of the two acids were comparable, presumably this factor did not account for the difference in acid stability. At the present 25% higher price for the coated product, its use rather than USP ascorbic acid would be justified if wheat flours were to be stored above 37°C at a moisture content of about 13.7%.

Destruction rates of brand B in wheat flour stored at 13.7% moisture are also included in Table I. Brand B was much more stable than brand A when storage was at 45°C, and somewhat more stable at 37°C. Both brands exhibited excellent stability at 26°C. In similar experiments with wheat flour at 12.9% moisture, no substantial difference in stability of the two USP acids was shown at the three test temperatures. Better storage stability at 45°C and 13.7% moisture exhibited by brand B over corresponding brand A might be related to particle size of the acids; brand B, showing better stability, was much coarser than brand A.

Results with CSM

Retention of brand A in CSM after storage is given in Fig. 2 and corresponding destruction rates in Table I. Large increases in rate of acid destruction occurred at both 37° and 45°C as moisture increased from 8.0 to 11.8%. Brand A was reasonably stable at 8.0% moisture at all three test temperatures. For good retention of ascorbic acid, CSM should contain not more than about 8.0% moisture if storage is to be as high as 45°C. From an Arrhenius-type plot, and a subsequent plot of destruction rate against moisture content, it was indicated that CSM at 9% moisture had good stability when stored at 40°C—70% retention after 16 weeks of storage. The maximum moisture content permitted at present under USDA specifications is 10.0%
(3); at this moisture content calculations indicated that CSM could lose about 80% of its ascorbic acid activity when stored for 3 months at 45°C. and about 40% at 37°C., but less than 10% at 26°C.

Destruction rates obtained when coated acid from the same manufacturer as brand A was used in CSM at the three moisture levels are also listed in Table I. These data show that coating markedly improved stability of ascorbic acid at all three storage temperatures when CSM contained 11.8 or 10.4% moisture. With CSM having 8.0% moisture, coated ascorbic acid had somewhat better stability at 45°C. compared with uncoated brand A, but at the lower temperatures both acids were stable.

Using coated acid rather than brand A would be desirable if CSM containing more than 8% moisture is to be stored at 37°C. or higher. However, even the coated acid is somewhat unstable in CSM at 10% moisture at 45°C., losing about half its potency in 16 weeks. When CSM has 8.0% moisture, a level frequent in commercial production, use of coated ascorbic acid at any probable average storage temperature could not be justified economically at present price differentials.

Results with Precooked Infant Cereals

Retention of ascorbic acid in precooked infant cereals after storage is shown in Fig. 3, and destruction rates are given in Table I. In these tests brand B was added to the cooked cereal before it was drum-dried. Destruction of the acid was quite rapid in all tests, the rate of destruction increasing with increases in moisture and temperature. Even the sample stored at 5.0% moisture at 26°C. retained only 62% acid after 10 weeks' storage. Figure 3 shows clearly the importance of storing this material at low moisture content and low temperature, and of maintaining these storage conditions with the help of good packaging, if a reasonable amount of the ascorbic acid is to be retained.

Also shown in Table I are destruction rates of ascorbic acid when brand B was dry-mixed with the cereal after it had been cooked, drum-dried, and ground. The dry-mix procedure gave results much improved over those obtained when the acid was added to the cook before drum-drying. At a normal shelf storage for the dry-mix material at about 7% moisture and not above 37°C., calculations indicate that at least 75% of the acid would be retained after 6 months of storage.

Summary and Conclusions

Ascorbic acid in wheat flour exhibited poor stability when the flour was stored at 14.6 or 13.7% moisture at 45°C. but was stable at these moisture levels at 26°C. Use of coated ascorbic acid (approximately 1% ethyl cellulose) from one manufacturer rather than the corresponding USP acid in wheat flour at 13.7% moisture resulted in much better storage stability at 45°C. Comparison of products from two manufacturers showed marked differences in stability under unfavorable storage conditions, with best stability from a coarser product.

Ascorbic acid in CSM was destroyed rapidly during storage at 37° and 45°C. when the blend contained 10.4 or 11.8% moisture, but the acid was reasonably stable at 45°, 37°, or 26°C. in CSM containing 8.0% moisture. When coated ascorbic acid was used instead of the corresponding USP acid, stability was improved markedly at 45° or 37°C. when the CSM contained 11.8 or 10.4% moisture, and some improvement was also obtained at 26°C. Only small improvements were noted at 8.0% moisture, both types of ascorbic acid being quite stable.

Ascorbic acid added to precooked infant cereal before drum-drying was destroyed rapidly when the blend was stored at a moisture content above 5.0% or when storage temperature was above 26°C. Even with storage at 26°C. and 5.0% moisture, retention was only about 62% after 10 weeks' storage. Stability was much improved when the acid was dry-mixed with a drum-dried and ground cereal, 75% retention of the ascorbic acid being indicated after 6 months' storage at 37°C. and 7% moisture.

Rate of destruction of ascorbic acid during storage of these food blends always increased as moisture content or temperature increased. The maximum moisture content for good acid stability at a given temperature differed for each material. Ascorbic acid in wheat flour stored at 13.7% moisture was reasonably stable, whereas the moisture content of CSM had to be no more than about 9% for similar stability. Ascorbic acid in infant cereal in which the acid was dry-mixed with the finished cereal at about 10% moisture showed similar acid stability. Ascorbic acid in infant cereal in which the acid was added before drum-drying was much less stable, even when stored at 5% moisture, and exhibited poor stability at higher moisture levels.

Use of coated ascorbic acid in dry blends with wheat flour or CSM could be justified economically for some storage conditions involving high moisture and temperatures.

Literature Cited

1. UNITED STATES DEPARTMENT OF AGRICULTURE, ASCS. Revised notice to corn-soya·milk manufacturers. Oct. 10, 1968 (mimeo.).

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