TOTAL ASH DETERMINATION IN SPICES

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The chemical determination most widely used by chemists in passing upon the quality of finely ground spices is that of total ash. This is best obtained by igniting one or two grams of the material, contained in a porcelain or platinum crucible, in a muffle furnace at the lowest temperature that will give a carbon-free residue. Heating for one hour at approximately 700° C. is usually sufficient. Red peppers require a little higher temperature than other spices to obtain the same result. Care must be exercised not to use a higher temperature than a dull red heat, or decomposition of carbonates in the ash will result.

Water-soluble and hydrochloric-acid-insoluble ash are determined usually only when the total ash or microscopic examination arouses suspicions of impurity or adulteration. If the total ash is low for the spice under consideration and the water-soluble ash very low the spice has probably been extracted. If the reverse is true of the total and acid-insoluble ash the spice contains extraneous mineral matter.

The color and appearance of the ash often tell something about the spice from which it was derived. Pure red peppers give a light greenish-blue ash, which is due to the presence of copper. American saffron or safflower has a red-brown ash; Spanish saffron yields a white or very pale gray ash; cinnamon ash is white or nearly so, and that of cassia is brown or brownish gray; clove ash is dark green, while most other kinds are white or gray. If spice ash is rubbed between the fingers the presence of foreign mineral matter in the original will be evidenced by a gritty feeling.

In spite of the simplicity of a total ash determination, the figures reported by different investigators vary considerably. A survey of the literature reveals the following partial list of figures as the average percentage of total ash in pure ginger: 4.39, 5.36, 5.55, 6.01, 3.80, 5.27, 3.62, 6.78, 3.66, 5.88, and 4.46. The first four are averages of more than 50 determinations each. Similar variations occur in results reported for other spices. This is chiefly due to variation in the spice itself and to a less extent to the personal equation.

Richardson (89) gives the total ash in Acheen black pepper as 8.99 per cent and that of Singapore black pepper as 5.41 per cent. These peppers are obtained from the same botanical species and the difference is largely due to variations in the circumstances surrounding their production, such as soil, climate, and handling.

Almost as large differences occur in spices from the same source from year to year as in those from different sources. This is due to differences in climatic conditions and has been well shown by Sindall (11). Table I has been prepared from some of his figures, each of which is the average of a large number of determinations upon pure cinnamon, imported from the same sources in successive years.

<table>
<thead>
<tr>
<th>Table I.—Per cent of ash in cinnamon</th>
</tr>
</thead>
<tbody>
<tr>
<td>1908</td>
</tr>
<tr>
<td>China cinnamon</td>
</tr>
<tr>
<td>Batavia cinnamon</td>
</tr>
</tbody>
</table>

In 1908 the average ash content of all samples from China was higher and in 1911 lower than that of Batavia samples in any year. The averages of the best figures available for four different varieties of cinnamon come between 4 and 4.15 per cent.

This variation is further shown by the percentages following which are the averages presented by numerous investigators for different varieties of black pepper.
Similar results have been reported for other spices. It seems useless, therefore, to consider the geographical source of a spice in interpreting a total ash determination except where a consistent difference is well authenticated. Sage seems to be one of the few such spices, for the American-raised variety appears to show consistently a higher percentage of ash than the Austrian.

While numerous figures are available for the percentages of ash in the commoner spices, few reliable results are published for the spices principally used by manufacturers, such as dill, turmeric, fennel, etc.

In order to draw conclusions from a total ash determination, it is desirable to know what percentage should be present when all factors tending to vary it have been eliminated. There are no such figures available at the present time. Each text book and article giving results of ash determinations reports a different figure for the same spice. This is not at all surprising when we consider that most of these figures are averages obtained with a few samples purchased at the same time and in the same market. When we remember further that different degrees of heating will result in different weights of ash from the same sample we understand why some chemists report twice as much ash as others for the same spice.

The percentages for total ash given in Table III are the result of an effort to find the amount of ash in the spices listed when all the conditions affecting

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**Table II.—Per cent of ash in different varieties of black pepper**

<table>
<thead>
<tr>
<th>Variety</th>
<th>Batavia</th>
<th>Acheen</th>
<th>Trang</th>
<th>Singapore</th>
<th>Tellicherry</th>
<th>Penang</th>
<th>Allepo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Per cent</td>
<td>4.93</td>
<td>10.90</td>
<td>5.17</td>
<td>4.56</td>
<td>6.44</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

**Table III.—Total ash determinations of various spices**

<table>
<thead>
<tr>
<th>Air-dried spice</th>
<th>Number of samples</th>
<th>Number of analysts</th>
<th>Per cent of ash</th>
<th>Per cent of ash</th>
</tr>
</thead>
<tbody>
<tr>
<td>Allspice</td>
<td>296</td>
<td>15</td>
<td>4.51</td>
<td>6</td>
</tr>
<tr>
<td>Anise</td>
<td>32</td>
<td>6</td>
<td>6.85</td>
<td>9</td>
</tr>
<tr>
<td>Bay leaves</td>
<td>29</td>
<td>5</td>
<td>4.63</td>
<td>8</td>
</tr>
<tr>
<td>Caraway</td>
<td>51</td>
<td>7</td>
<td>6.73</td>
<td>10</td>
</tr>
<tr>
<td>Cardamom fruit</td>
<td>109</td>
<td>12</td>
<td>6.71</td>
<td>8</td>
</tr>
<tr>
<td>Cinnamon</td>
<td>143</td>
<td>4</td>
<td>4.28</td>
<td>5</td>
</tr>
<tr>
<td>Cloves</td>
<td>101</td>
<td>4</td>
<td>8.35</td>
<td>10</td>
</tr>
<tr>
<td>Coriander</td>
<td>51</td>
<td>7</td>
<td>4.63</td>
<td>8</td>
</tr>
<tr>
<td>Coriander</td>
<td>270</td>
<td>4</td>
<td>7.63</td>
<td>10</td>
</tr>
<tr>
<td>Cumin</td>
<td>51</td>
<td>7</td>
<td>8.12</td>
<td>10</td>
</tr>
<tr>
<td>Fennel</td>
<td>48</td>
<td>7</td>
<td>6.15</td>
<td>7</td>
</tr>
<tr>
<td>Ginger</td>
<td>841</td>
<td>20</td>
<td>4.89</td>
<td>5</td>
</tr>
<tr>
<td>Mace</td>
<td>210</td>
<td>10</td>
<td>4.26</td>
<td>3</td>
</tr>
<tr>
<td>Marjoram</td>
<td>156</td>
<td>5</td>
<td>10.62</td>
<td>10</td>
</tr>
<tr>
<td>Mustard</td>
<td>151</td>
<td>21</td>
<td>4.83</td>
<td>5</td>
</tr>
<tr>
<td>Mustard flour</td>
<td>48</td>
<td>7</td>
<td>5.49</td>
<td>6</td>
</tr>
<tr>
<td>Nutmeg</td>
<td>342</td>
<td>8</td>
<td>2.65</td>
<td>5</td>
</tr>
<tr>
<td>Onion</td>
<td>48</td>
<td>7</td>
<td>4.28</td>
<td>5</td>
</tr>
<tr>
<td>Paprika</td>
<td>449</td>
<td>12</td>
<td>6.78</td>
<td>5</td>
</tr>
<tr>
<td>Pepper, black</td>
<td>581</td>
<td>22</td>
<td>5.03</td>
<td>7</td>
</tr>
<tr>
<td>Do...cayenne</td>
<td>237</td>
<td>15</td>
<td>6.17</td>
<td>7</td>
</tr>
<tr>
<td>Do...other red varieties</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Do...white</td>
<td>660</td>
<td>13</td>
<td>1.26</td>
<td>3.5</td>
</tr>
<tr>
<td>Safflower</td>
<td>18</td>
<td>4</td>
<td>4.67</td>
<td>7</td>
</tr>
<tr>
<td>Saffron</td>
<td>156</td>
<td>14</td>
<td>5.36</td>
<td>7</td>
</tr>
<tr>
<td>Sage</td>
<td>311</td>
<td>2</td>
<td>7.39</td>
<td>10</td>
</tr>
<tr>
<td>Savory</td>
<td>29</td>
<td>4</td>
<td>9.94</td>
<td>7</td>
</tr>
<tr>
<td>Star anise</td>
<td>63</td>
<td>3</td>
<td>2.67</td>
<td>5</td>
</tr>
<tr>
<td>Thyme</td>
<td>104</td>
<td>4</td>
<td>9.83</td>
<td>5</td>
</tr>
<tr>
<td>Turmeric</td>
<td>36</td>
<td>9</td>
<td>6.37</td>
<td>11</td>
</tr>
</tbody>
</table>
Total Ash Determination in Spices

it had been averaged. A list was first made of every available average obtained with goods known to be pure, for each kind of spice. Such figures were obtained from most of the articles listed in the bibliography. A few results which were clearly unreliable were then eliminated, and weighted averages prepared from those remaining. The determinations upon which they are based were made by many different analysts over a period of forty years. Many of them have not previously been published. The unpublished determinations, numbering several thousand, were made chiefly by James Blaine Martin, for the use of whose results grateful acknowledgment is hereby made, and by the author, in the Meat Inspection Laboratory of the United States Department of Agriculture. All the samples were examined physically and microscopically, and no determination on questionable material was used in the preparation of this table.

Variations from these figures should not exceed one-third of their value. For comparison there is included in this list the maximum total ash permissible by the Bureau of Chemistry in the enforcement of the pure food laws.

The approximate ash content of other spices used to some extent in the powdered form is given in Table IV. It is not claimed that these figures are representative, for they are averages of only a few determinations in each case. However, inasmuch as there are no others available, they are given for whatever value they may have.

The approximate composition of the ash derived from several spices is shown by the composite analyses tabulated below.

<table>
<thead>
<tr>
<th>Table IV.—Approximate ash content of spices named</th>
</tr>
</thead>
<tbody>
<tr>
<td>Per cent</td>
</tr>
<tr>
<td>Basil</td>
</tr>
<tr>
<td>Calamus</td>
</tr>
<tr>
<td>Capers</td>
</tr>
<tr>
<td>Cassia buds</td>
</tr>
<tr>
<td>Charlock</td>
</tr>
<tr>
<td>Dill</td>
</tr>
<tr>
<td>Fengreek</td>
</tr>
<tr>
<td>Garlic</td>
</tr>
<tr>
<td>Juniper berries</td>
</tr>
<tr>
<td>Parsley</td>
</tr>
<tr>
<td>Tarriac</td>
</tr>
<tr>
<td>Vanilla beans</td>
</tr>
</tbody>
</table>

| Table V.—Approximate composition of ash from spices named |

<table>
<thead>
<tr>
<th></th>
<th>Black pepper</th>
<th>White pepper</th>
<th>Mustard</th>
<th>Papi ka</th>
<th>Cinamon</th>
<th>Cassia</th>
<th>Cardasmon</th>
<th>Marjoram</th>
<th>Fenugreek</th>
</tr>
</thead>
<tbody>
<tr>
<td>K₂O</td>
<td>27.56</td>
<td>6.13</td>
<td>18.90</td>
<td>54.37</td>
<td>14.23</td>
<td>5.55</td>
<td>10.32</td>
<td>19.22</td>
<td>33.20</td>
</tr>
<tr>
<td>Na₂O</td>
<td>3.89</td>
<td>0.79</td>
<td>0.37</td>
<td>3.98</td>
<td>4.02</td>
<td>0.91</td>
<td>20.01</td>
<td>67.69</td>
<td>5.51</td>
</tr>
<tr>
<td>CaO</td>
<td>13.73</td>
<td>32.07</td>
<td>15.57</td>
<td>5.15</td>
<td>39.02</td>
<td>51.30</td>
<td>13.20</td>
<td>20.05</td>
<td>8.57</td>
</tr>
<tr>
<td>MgO</td>
<td>7.55</td>
<td>10.58</td>
<td>10.51</td>
<td>6.02</td>
<td>3.35</td>
<td>1.19</td>
<td>4.56</td>
<td>5.67</td>
<td>7.10</td>
</tr>
<tr>
<td>FeO₂</td>
<td>0.88</td>
<td>2.04</td>
<td>1.09</td>
<td>1.97</td>
<td>0.48</td>
<td>6.11</td>
<td>3.22</td>
<td>6.68</td>
<td>2.33</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>0.20</td>
<td>0.55</td>
<td>0.37</td>
<td>0.75</td>
<td>0.18</td>
<td>5.05</td>
<td>0.65</td>
<td>0.10</td>
<td></td>
</tr>
<tr>
<td>SiO₂</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Cl</td>
<td>9.13</td>
<td>1.7</td>
<td>3.51</td>
<td>0.56</td>
<td>0.11</td>
<td>2.35</td>
<td>1.76</td>
<td>4.97</td>
<td></td>
</tr>
<tr>
<td>CO₂</td>
<td>12.90</td>
<td>14.81</td>
<td>2.82</td>
<td>31.55</td>
<td>31.02</td>
<td>4.51</td>
<td>7.26</td>
<td>10.66</td>
<td></td>
</tr>
<tr>
<td>SO₂</td>
<td>6.56</td>
<td>3.55</td>
<td>6.79</td>
<td>2.88</td>
<td>0.39</td>
<td>0.84</td>
<td>20.37</td>
<td>24.20</td>
<td>4.66</td>
</tr>
</tbody>
</table>
SUMMARY

1. The ash content of spices is affected by numerous factors, so that analyses made upon samples derived from a particular source and crop are not likely to be representative for that spice in general.

2. Figures representing the percentage of total ash which should normally occur in practically all spices in general use have been presented.

3. Composite analyses of the ash of several spices are given.

BIBLIOGRAPHY

ALSPICE


CALAMUS


CARDAMOM


CLOVES


CUMIN


FENNEL


FENUGREEK


GINGER

MACE

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(43) Windisch, C. H., and Piesse, A. E.

MARJORAM

(41) Kopp, G.

(42) Spafford, W.

(43) Windisch, R.

MUSTARD

(44) Piesse, C. H., and Stansell, L.

(45) Walker, E., and Martin, E. W.

(46) Drew, C. W.

(47) Leach, A. E.

(48) McGill, A.

(49) McGill, A.

NUTMEG

(50) Buss, W.

(51) Ranwez, F.

(52) Vanderplanken, J.

PAPRIKA

(53) Bittó, B. von.

(54) Origo, O.

(55) Windisch, C. H.

(56) Windsor, F.

(57) Stillwell, A. G.

(58) Doolittle, R. E., and Ogden, A. W.

PARSLEY

(59) Dahlen, H. W.

(60) Massute, F.

PEPPER, BLACK AND WHITE

(61) Lentz, W.

(62) Heisch, C.

(63) Johnstone, W.

(64) Bimbi, F.

(65) Doolittle, R. E.

(66) Hutton, L.

(67) McGill, A.

(68) McGill, A.

(69) McGill, A.

(70) McGill, A.

(71) McGill, A.

(72) McGill, A.

(73) McGill, A.

(74) McGill, A.

PEPPER, RED AND CAYENNE

(75) Kynaston, W. C. R.

(76) Lenton, W. H.

(77) Sindall, H. E.

(78) Tolman, L. M., and Mitchell, L. C.

(79) Boyles, F. M.

SAFFRON

(80) Kunte, G., and Hillger, A.

(81) Galliou, M.

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(83) Krizan, R.