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Determining the Metal Content of Cotton

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Metals in cotton may contribute to problems that occur in yarn manufacturing, fabric production, bleaching and dyeing processes.[1-4] High levels of silicon and other metals have been suspected as a cause of frictional problems in rotor spinning and needle wear in knitting. In the dyeing of cotton, insoluble calcium and magnesium salts may interfere with dyeing quality.[3] Copper, iron and aluminum contribute to yellowness of finished denim goods.[4] Proper disposal of solid and liquid textile wastes containing metals and residual metals in commercial fabrics are problems of increasing importance to the textile industry.[5] Environmental limits on certain metals are strict and are expected to be even stricter in the future.

ABSTRACT

Metal content of cotton is of increasing importance to cotton processors both because of effects on processing and product qualities and because of potential environmental concerns and constraints. Metals can contribute to problems that occur in yarn manufacturing, fabric production, bleaching and dyeing processes. Disposal of both liquid and solid wastes containing certain metals must be carefully monitored and controlled. Cottons varying in growth area, variety and harvest season were analyzed for potassium, magnesium, calcium, sodium, iron, copper, manganese, zinc, lead and cadmium contents by atomic absorption spectroscopy. Metal contents varied significantly between years and between growth areas. Potassium was the dominant metal (2000-6500 ppm) followed by magnesium and calcium (400-1200 ppm), sodium (100-300 ppm), iron (20-90 ppm) and zinc, manganese and copper (1-10 ppm). Lead and cadmium were not detectable in any of the cottons at test sensitivity limits of about one ppm.

KEY TERMS

- Ash Content
- Atomic Absorption Spectroscopy (AA)
- Cotton
- Inductively Coupled Plasma Emission Spectroscopy (ICP)
- Metal Content

Naturally occurring metals such as potassium are important nutrients for normal development of cotton.[6] The potassium in cotton is in a very soluble form and residual levels in cotton at the time of harvest are highly related to field weathering conditions.[7,8] Other metals such as aluminum, calcium, magnesium, iron, silicon and sodium also occur naturally in cotton, but in smaller amounts. Trace amounts of copper, manganese and zinc also are often present.[9,10]

There is little information available on the factors that cause variations in metal content, but factors such as variety, cultural and harvesting histories, location and field weathering conditions are obviously important. Metal compositions of domestic cottons covering the years 1983 to 1989 were determined. To determine regional and varietal differences, four varieties grown in three locations in one year were tested. Three African, three Chinese and one Pakistani cottons were also tested. The metals determined were potassium, calcium, magnesium, sodium, iron, copper, manganese, zinc, cadmium and lead.

Experimental

Cottons from inventory were conditioned in the laboratory atmosphere (24C and 62% RH) for at least 20 days before moisture contents were determined for each. Moisture contents (in triplicate) for the cottons were determined and averaged 6.30 ± 0.26%. Metal contents were calculated on a dry fiber basis, using this 6.3% moisture content as a factor to adjust to dry weight.

Duplicate 10 ± 0.005 g samples of raw cotton were ashed for 2.5 hours at 650C in a muffle oven. After ashing, the residues were weighed and ash content was calculated for each sample. The ash residues were dissolved in 5 mL of concentrated hydrochloric acid (38%) and subsequently made up to 25 mL with deionized water. Dilutions were made from this original solution with deionized water depending on the expected concentration of the element in the sample and the linear operating range of the atomic absorption instrument as predetermined with standard solutions.

Atomic absorption (AA) analysis for each metal was made on a Buck Model 200A Spectrophotometer[a] reading in the absorbance mode using an air/acetylene flame. To overcome any possible ionization of the potassium or sodium in the air/acetylene flame, cesium at the concentration of 1000 µg/mL of sample and standard was added when determining these two elements. The cesium was supplied by dissolving 3.1675 g of cesium chloride in 5 mL of deionized water. Additions of the cesium chloride to standards and samples were at the rate of 2 µL/ml. To overcome possible depression of the sensitivity for calcium and magnesium by silicates, aluminum or phosphates, 1000 µg/mL lanthanum was added to standards and samples when analysis for these two elements were performed. The lanthanum solution was prepared by dissolving 6.6840 g of lanthanum chloride in 5 mL of deionized water. Two microliters of the lanthanum solution per milliliter of sample were added.

Dilutions made to obtain effective absorbencies at their respective wavelengths in linear operating ranges were 1250x, 625x and 125x total for potassium, calcium and magnesium and sodium, respectively. The original 10 grams per 25 mL of solution (2.5x) was

[a] Trade names are used solely to provide information. Mention of a trade name does not constitute a warranty or an endorsement of the product by the U.S. Department of Agriculture to the exclusion of other products not mentioned.

Table I. Comparison of ICP and AA for Certain Metals in Raw Cotton

Cotton	Ash %	K ICP	K AA	Ca ICP	Ca AA	Mg ICP	Mg AA	Fe ICP	Fe AA	Cu ICP	Cu AA	Mn ICP	Mn AA	Zn ICP	Zn AA
		Metal Content (ppm)													
So. Carolina	1.26	4600	4170	500	735	700	665	20	19	1	1	3	4	5	5
California	1.71	5400	5580	700	565	600	600	46	55	3	4	5	5	12	7
Texas	1.04	3800	3440	600	525	400	345	15	22	1	2	3	3	5	4
Mississippi	1.16	3700	3680	900	930	600	505	50	49	1	3	5	5	6	5
Texas High Plains	1.75	5300	5680	1000	1000	600	585	60	50	1	2	6	5	6	6
Average		4650	4510	740	751	580	540	38	39	1.4	2.4	4.4	4.4	6.8	5.4

used for determination of copper, iron, manganese, zinc, lead and cadmium. At least duplicate dilutions and determinations were made on each sample and calculations of concentration were based on a calibration curve using a minimum of four standard concentrations. A blank, no ash, was always included in each determination and used as a reference sample.

Five cottons were selected—one each from California, Mississippi, Texas High Plains, Texas and South Carolina—and submitted to the Plant Tissue Analysis Laboratory at Clemson University for Inductively Coupled Plasma Emission Spectroscopy (ICP) analysis. Single analyses for potassium, calcium, magnesium, copper, manganese, iron and zinc were determined for each of the five cottons. Cottons were also analyzed in our laboratory by the AA procedure detailed above, except that in the case of copper, triplicate measurements were made on duplicate ash samples.

Results and Discussion

Potassium, magnesium, calcium, sodium, iron, manganese, zinc and copper were present in all cottons tested. Neither lead nor cadmium could be detected in any of the cottons at the test sensitivity limits of 0.5 ppm for cadmium and 2 ppm for lead.

Comparison of ICP and AA Analysis

The results of ICP and AA tests for metals in five cottons show that agreement between the two methods and the two laboratories is good, see Table I. When comparing the ICP and AA methods for metals present at levels greater than 10 ppm (potassium, calcium, magnesium, iron) the results satisfy the T-test for equal means and variances at the 95% confidence level. Even though statistical methods were not used to show differences between the methods for the metals (copper, manganese and zinc) present at low levels, the agreement between methods appears good.

For the five cottons, ash contents were lowest for the Texas, Mississippi and South Carolina cottons and highest for the Texas High Plains and California cottons ranging from a low of 1.04% for the Texas cotton to a high of 1.75% for the Texas High Plains cotton.

Potassium levels ranged from about 3500 ppm for the Texas and Mississippi cottons to a high of about 5700 ppm for the Texas High Plains and California cottons. Calcium levels ranged from about 500 to 1000 ppm with the higher levels appearing in Mississippi and Texas High Plains cottons. Magnesium level was lower for the Texas cottons than for the other cottons. Iron levels were below 100 ppm for all cottons. Copper, zinc and manganese were present in each of these cottons at low levels.

The Effect of Area of Growth and Variety

Several different varieties grown in 1983 in three domestic growing areas were tested. The varieties were SJ5, DPL61, GSA71 and SJC grown in California and SJ5, DPL61 and GSA71 grown in Texas and Mississippi. In each area, each variety was grown in the same field. Results are shown in Table II. Potassium content of cottons grown in California was greater than 5300 ppm for all four varieties. The SJC and SJ5 varieties contained 6600 and 6200 ppm, respectively. The levels in the GSA71 and DPL61 varieties were slightly lower at 5300 and 5700 ppm, respectively. Considerably less potassium was found in these same varieties grown in Mississippi and Texas. In Mississippi the three varieties averaged 3000 ppm and in Texas about 2200 ppm. In each area, potassium content of the SJ5 was higher than that of either the DPL61 or the GSA71 grown in that same area. Average potassium levels of cottons grown in California were twice as high as those of the cottons grown in Mississippi and about three times as high as those of the cottons grown in Texas. Potassium contents of the foreign cottons ranged from about 4300 to 5500 ppm. The averages were 4700 ppm for the African cottons, 5200 ppm for the Chinese cottons and 5300 ppm for the single Pakistani cotton.

Calcium content ranged from about 500 to 800 ppm for the four domestic varieties in the three locations. Differences between varieties were small. However, the levels in cottons grown in Texas averaged about 750 ppm whereas levels in cottons grown in California and Mississippi averaged

Table II. The Effect of Growing Area and Variety on Metal Content of Raw Cotton

Growing Area	Variety	K	Ca	Mg	Na	Fe	Cu	Mn	Zn
		Metal Content (ppm)							
California	SJC	6610	520	660	145	15	2	3	5
	SJ5	6230	550	670	190	21	3	4	7
	GSA71	5660	485	495	140	16	2	3	4
	DPL61	5340	645	505	135	17	3	4	5
Mississippi	SJ5	3850	555	445	115	40	2	3	4
	GSA71	2120	530	375	160	31	2	4	4
	DPL61	3030	550	345	105	36	2	4	4
Texas	SJ5	2240	795	445	110	93	3	7	4
	GSA71	2160	810	380	110	84	3	7	6
	DPL61	2120	680	380	105	82	2	7	5
	I. D. Mark								
China	229	5440	810	760	340	33	2	5	5
	329	4970	695	630	285	22	2	3	4
	527	5120	575	585	100	30	2	5	8
Africa	BOLA	5220	575	695	175	43	2	8	4
	LIBA	4330	1175	545	190	82	2	7	3
	VIVA	4580	695	585	145	63	3	8	5
Pakistan	ORGINE	5280	1525	695	270	15	1	0	1

about 550 ppm. Calcium levels in the foreign cottons were somewhat higher than the levels in domestic cottons.

Magnesium contents ranged from about 350 to 675 ppm for the domestic cottons. As was the case with potassium, the SJ5 variety contained slightly higher levels of magnesium than did the other varieties grown in the same areas. Cottons grown in California averaged 580 ppm which was approximately 50% higher than the average for the cottons grown in Texas and in Mississippi. Magnesium contents of the foreign cottons were slightly higher than that of the domestic cottons.

Sodium contents varied from 100 to 190 ppm for the domestic cottons. No significant differences were found between varieties. The cottons grown in California had slightly higher levels than those grown in Mississippi or Texas. Sodium contents of the foreign cottons were slightly higher than those of the domestic cottons.

Iron contents of all domestic cottons were below 100 ppm. The cottons grown in Texas averaged about 85 ppm, those grown in Mississippi averaged between 30 and 40 ppm and those grown in California averaged between 15 and 25 ppm. Differences between varieties were small. Iron contents of the foreign cottons were also below 100 ppm.

Average levels of zinc and manganese in the domestic cottons were about 5 ppm. The manganese levels of the cottons grown in Texas appeared to be slightly higher than the levels in the cottons grown in California or Mississippi. Copper levels were generally between 2 and 3 ppm for most of the cottons. The Pakistani cotton had very low levels of copper and zinc and no detectable manganese. The results for copper and manganese agree generally with results reported by Heinzelman.[10]

The Effect of Growing Location and Season

Metal contents of cottons grown between 1983 and 1989 in four locations are shown in Table III. Not all years are represented in any one area. Averages and ranges of potassium contents of cottons from the different areas and years were—California, average 5853 ppm, range 4800-6400 ppm; Mississippi, average 3612 ppm, range 3000-4400 ppm; Texas, average 3508 ppm, range 2170-5170 ppm; and Texas High Plains, average 5107 ppm, range 3510-6130 ppm. In general, the levels in California cottons were consistently high and the levels in the Mississippi cottons were consistently low. The levels in the Texas and the Texas High Plains cottons were more variable than the levels in cottons from the other areas.

Table III. Potassium, Calcium, Magnesium, Sodium and Iron Contents of Raw Cottons from Different Growing Areas in Different Years

Growing Area	1983	1984	1986	1987	1988	1989
			Potasium (ppm)			
California	5960	5980	6150	5810	4820	6400
Mississippi	3000	—	4390	4030	3160	3480
Texas	2170	—	3600	3090	5170	—
Texas High Plains	—	—	3510	6130	5680	—
			Calcium (ppm)			
California	550	965	855	910	650	780
Mississippi	545	—	1145	1080	1250	860
Texas	760	—	960	1235	1025	—
Texas High Plains	—	—	1035	750	935	—
			Magnesium (ppm)			
California	585	555	735	685	695	610
Mississippi	390	—	1145	1235	1250	570
Texas	400	—	395	395	500	—
Texas High Plains	—	—	465	545	585	—
			Sodium (ppm)			
California	150	195	225	170	220	250
Mississippi	125	—	135	215	170	135
Texas	110	—	195	155	185	—
Texas High Plains	—	—	140	160	175	—
			Iron (ppm)			
California	18	40	66	38	60	29
Mississippi	36	—	26	26	35	29
Texas	86	—	21	38	35	—
Texas High Plains	—	—	60	55	50	—

The calcium content of the cottons averaged over areas and years was 905 ppm and the range was 545-1250 ppm. There were no clear effects related either to area or to year of growth.

Averages and ranges of magnesium contents of cottons from the different areas and years were—California, average 644 ppm, range 555-735 ppm; Mississippi, average 918 ppm, range 390-1250 ppm; Texas, average 423 ppm, range 395-500 ppm; and Texas High Plains, average 532 ppm, range 465-585 ppm. For the most part, magnesium contents were relatively consistent over years and areas and fell within a range of about 400-700 ppm. Exceptions to this were the much higher levels in the 1986, 1987 and 1988 Mississippi cottons where average magnesium content was 1210 ppm.

The average sodium content of the cottons was 173 ppm. The levels were very consistent both between years and between growth areas. The iron contents of the cottons were generally in the range of 20-85 ppm and no patterns related either to year or to area of growth were apparent.

As was the case with the area of growth-variety cottons, the manganese, zinc and copper contents of these cottons were 10 ppm or below. Each of the same metals were present in each of the cottons tested. Manganese levels ranged from 4-8 ppm, zinc levels ranged from 3-10 ppm and copper levels ranged from 1-9 ppm. No differences were found that could be related either to year or to area of growth.

Cotton Ash Content

Ash content of raw cotton can vary from below one percent to well above two percent depending on factors including area of growth, environmental conditions, foreign matter content and others.[9] In combined studies of cottons from five domestic and three foreign growing areas, ash determinations were made on a total of 42 cottons, see Table IV.

Table IV. Ash Content of Cottons Grown Domestically and Internationally

Location	Ash %	Range %
California ($n = 13$)	1.80	1.61 - 2.07
Mississippi ($n = 10$)	1.17	0.79 - 1.42
Texas ($n = 7$)	1.07	0.91 - 1.71
Texas High Plains ($n = 4$)	1.62	1.41 - 1.90
South Carolina ($n = 1$)	1.26	—
Foreign ($n = 7$)	1.70	1.59 - 1.84

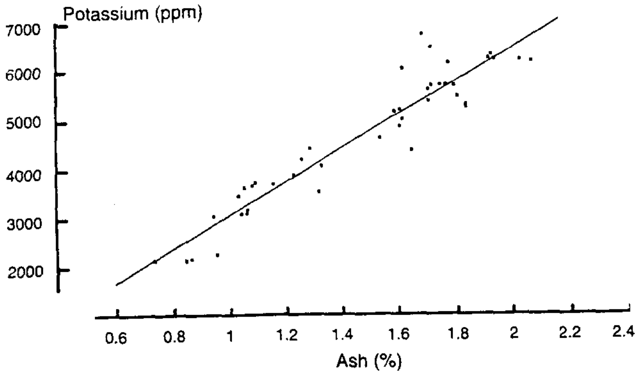

Fig. 1. The relationship between potassium and ash contents of cotton.

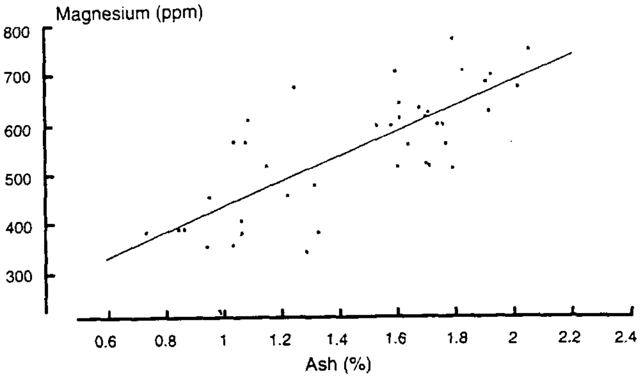

Fig. 2. The relationship between magnesium and ash contents of cotton.

These results fit into a somewhat typical pattern of higher levels of noncellulosic constituents in cottons that are grown in western areas where irrigation is the norm and rain on open bolls is rare. In general, the levels of noncellulosic constituents, including metals, are always high in California cottons relative to cottons from other major domestic growing areas.[2]

The relationship between potassium content and ash content of the 42 cottons is shown in Fig. 1. The coefficient of simple correlation is 0.94. The relationship between magnesium content and ash content is shown in Fig. 2. The coefficient of simple correlation is 0.76. When the potassium and magnesium contents are combined and related to ash content, the coefficient of simple correlation is 0.95. Thus, for the cottons tested, above 90% of the variation in ash content is accounted for by variations in potassium and magnesium contents. Potassium (31%), calcium (6%) and magnesium (4%) account for approximately 41% of ash weight in the cottons. The results are generally comparable to reported results.[9]

Summary and Conclusions

Ash and metal contents were determined for 42 cottons from major domestic growing areas and from three foreign countries. The basic procedure involved ashing of the cotton samples, dissolving the ash in acid solution and determination of the metals using AA. Selected samples were tested both by AA method and by ICP in an independent laboratory. Results of these comparison tests were very good.

The dominant metal in the cottons was potassium (ca., 2000-6500 ppm); followed by calcium and magnesium (ca., 400-1200 ppm); sodium (ca., 100-300 ppm); iron (ca., 20-90 ppm); and zinc, manganese and copper (ca., 1-10 ppm). All of the cottons tested had measurable quantities of these last three heavy metals. Cadmium and lead were not detectable in any of the cottons at the test sensitivity limits. The western grown, irrigated cottons, particularly those from California, generally had higher ash contents and higher levels of potassium than did cottons grown in the more easterly areas. There were large variations in metal contents both between years and between locations, particularly for potassium, magnesium and iron. The only varietal difference noted was with the SJ5 cotton. In direct comparisons in three growing areas, the potassium and magnesium contents of the SJ5 were always higher than that of either the DPL61 or GSA71 varieties. There was a high positive correlation between cotton ash content and combined potassium and magnesium content.

Although this is obviously not a complete survey of all metals for all variations of cotton—nor would one be possible—the cottons tested represent major growing areas over several seasons and the metals determined include most of the common ones found in significant quantities in cotton with several notable exceptions such as aluminum and silicon. Thus, these results should provide general information to cotton processors who have concerns about the roles of metals in yarn manufacturing, dyeing, finishing, safety and health in the workplace and disposal of solid and liquid wastes containing metals.

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