National Research Council

VALIDATION OF THE TEST METHOD FOR THE EXTRACTION OF CHROMIUM FROM DYED TEXTILES

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Chromium: **Why?**

Because:

1. it is one of the most abundant element present in nature
2. it exists in several oxidation states with very different toxicity
3. it is also used in different textile processes, as dyeing and printing, and it is present above all in metal complex dyes
4. its presence in textiles is not regulated by binding national or EU standards (except for some REACH regulations)

Cr III

- essential element for carbohydrate, normal protein and lipid metabolism, and for proper insulin function
- used as micronutrient and dietary supplement

Cr VI

- negative effects on environment and on organisms
- genotoxic and carcinogenic
- causes allergic dermatitis
- induces oxidative stress, DNA damages, apoptotic cell death and altered gene expression

It is very important to determine chromium content in textiles, for the safety of the workers and for the consumers too.

IMPORTANCE IN TEXTILE INDUSTRY

Chrome dyes:

- are cheap and easy to apply
- are level dyeing
- have excellent fastness properties

The use of dyes containing groups capable of forming chromium complexes when treated with potassium dichromate is declined.

They have been replaced to a large extent by metal-complex dyes, in which the chromium-dye complex is preformed during the dyestuff manufacturing.

National or European Standards about restriction on presence of chromium in textiles don't exist!

BUT

Some ecological voluntary schemes (Oeko – Tex Standard 100) require chromium limits based on the extraction of the metal by means of a saline solution simulating the human acid perspiration.

the saline solution concentration and the parameters applied refer to **ISO 105 E/04** on color fastness tests

ANALYTICAL PROBLEMS

✓ Absence of standardized analytical methods for chromium extraction

✓ Application of in-house or not validated methods by individual laboratories

inhomogeneous and not reproducible data

The method for the analytical determination of extractable chromium from textile products in a acid perspiration solution was investigated in depth:

➢ critical steps of the analytical procedure

➢ the best conditions of extraction for textiles dyed with a high-concentrated chromium dyestuff

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OBJECTIVES

1. *To verify the reproducibility and the sensibility of the method*

Application of the tested method to a textile material dyed with a low-concentrated chromium dyestuff, varying:

- ✓ **temperature**
- ✓ **time of contact**
- ✓ **liquor ratio**

2. *To verify the efficiency of the method by the determination of total chromium in the ashes*

Application of the UNI 8047 standard about the determination of total chromium in the ashes.

EXPERIMENTAL PARAMETERS

TEMPERATURE
- **30°C**
- **40°C**
- **50°C**

TIME OF CONTACT
- **1 h**
- **2 h**

LIQUOR RATIO
- **1 g :50 ml**
- **1 g :100 ml**

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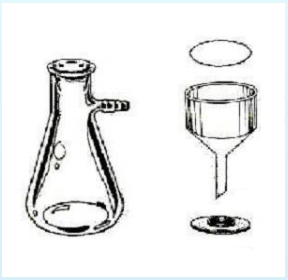
WOOL TOP

acid perspiration solution

filtration on vacuum



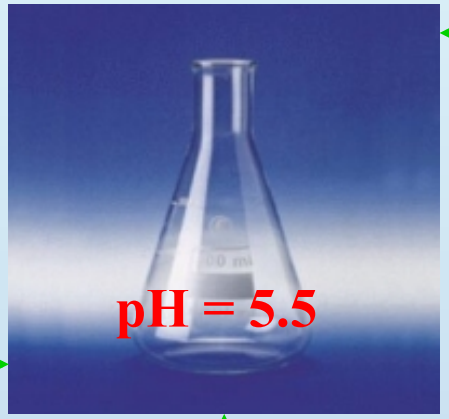
Total chromium determination by GFAA

Hexavalent chromium determination by UV-Vis

Acid saline solution: ISO 105 E/04 on colour fastness tests

distilled water

sodium dihydrogen
phosphate dihydrate



sodium chloride

chlorohydrate L-istidine
monohydrate

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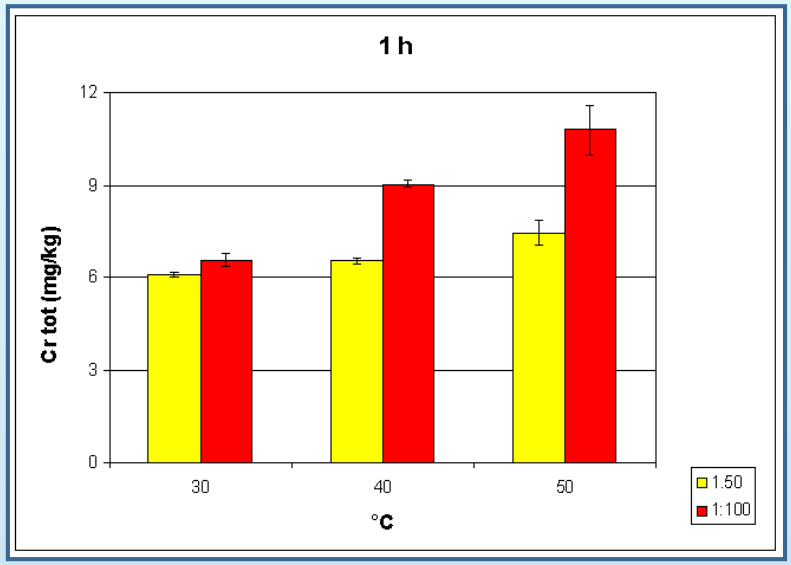
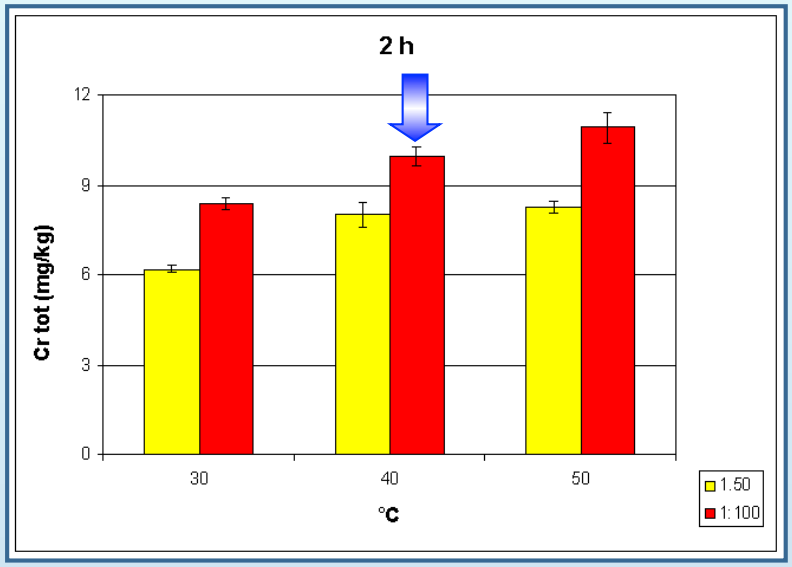
RESULTS

TEST	T (°C)	Liquor ratio	Concentration (mg/kg)							
			Total chromium				Hexavalent chromium			
			1 h	CV%	2 h	CV%	1 h	CV%	2 h	CV%
1	30°C	1: 50	6.09	1.6	6.19	1.6	0.98	4.1	1.14	8.8
2	30°C	1:100	6.56	3.0	8.36	2.4	1.39	7.2	1.87	5.3
3	40°C	1: 50	6.54	1.5	7.99	5.0	1.03	9.7	1.48	6.8
4	40°C	1:100	9.04	1.1	9.95	3.0	1.59	1.3	2.00	5.0
5	50°C	1: 50	7.44	5.4	8.25	2.4	1.44	6.9	2.36	8.5
6	50°C	1:100	10.78	7.4	10.92	4.6	2.38	1.7	2.63	7.6

Temperature, liquor ratio and time of contact affect the extraction in a significant way.

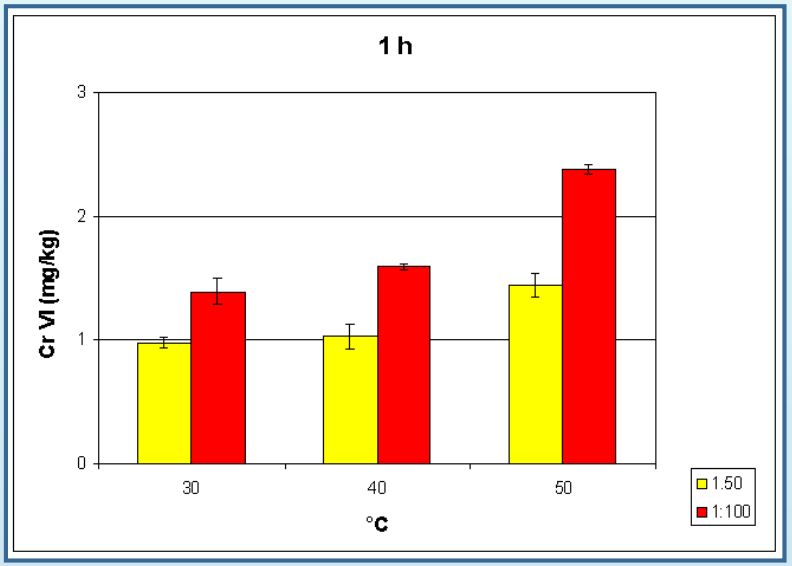
TOTAL CHROMIUM

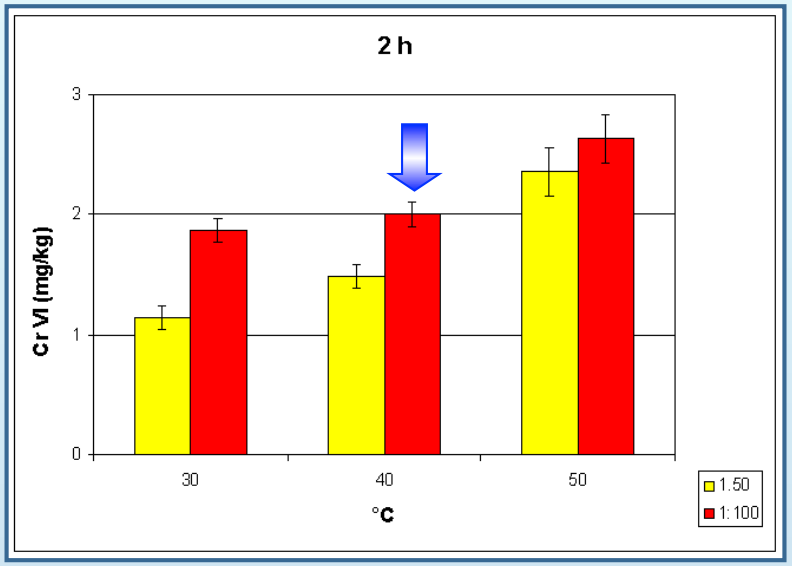




- Increasing the temperature: increase of the extractable metal content, but at 50°C data are less reproducible
- Increasing the liquor ratio: better extraction efficiency
- Increasing the time of contact: increase of the extractable metal content

HEXAVALENT CHROMIUM





- Cr VI extraction is affected positively by temperature, liquor ratio and time of contact.
- Cr VI results confirmed the Cr tot ones.

TOTAL CHROMIUM IN THE ASHES

UNI 8047

TEST	T (°C)	Liquor ratio	Yield (%)	
			1 h	2 h
1	30°C	1:50	28.1	28.6
2	30°C	1:100	30.3	38.6
3	40°C	1:50	30.2	36.9
4	40°C	1:100	41.7	45.9
5	50°C	1:50	34.3	38.1
6	50°C	1:100	49.8	50.1

The percentage of metal extraction yield vary according to the extraction conditions, from **28%** to **50%**.

CONCLUSIONS

- The extraction of chromium from textile products with an acid perspiration solution can vary according to different analytical conditions and it is affected by a low reproducibility

- The solubility of chromium into the saline solution constitutes the critical stage of the analytical method tested

- Increasing the temperature, the liquor ratio and the time of contact, chromium extraction is affected positively

- The conditions that lead to a better extraction efficiency with greater results reproducibility seem to be a temperature of 40°C, a liquor-ratio of 1:100 and a time of contact of 2 hours.

FUTURE AIMS

- To improve the study of the chemical-physical mechanism that controls the release of the metals from the textile materials to the saline extraction solution

- To apply the tested method to different heavy metals extraction from different type of textile materials

- To apply the selected conditions to the chromium extraction using an artificial skin surface film liquid (SSFL)

- To develop a definite analytical standard for the determination of heavy metals in textile products, and to propose it as the official analytical method

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THANKS

FOR THE ATTENTION!

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