A Technical Presentation in DIFSC2010

By
Dr. Alamuri Gopala Krishnamacharyulu
Principal Food Analyst,
Food Chemical Analysis unit
Food & Environment laboratory
Dubai Central Laboratory, DUBAI (U.A.E)

□2003 May: Sudan I detected in Chilli products (France)

□2003 July: The products contaminated with Sudan I in

France were later found out to be produced in

UK/produced in Italy and imported in to UK

□2005 February: A Worcester sauce was reported to be

contaminated with Sudan I. This

contamination led over 400 products being

taken off the shelves.

Cause of this contamination in many of these cases was linked to Chilli powder, chilli products or curry powder that had been illegally contaminated with Sudan dyes.

□2005 May:

Sixty nine products were with drawn from sale by companies in UK due to presence of Para Red.

 $\square 2005 - 2007$:

Sudan I traces were found in several spices (South Africa)

Safety of Sudan dyes

- Sudan dyes are suspected carcinogens
- IARC considers Sudan I,II,III,IV as Group 3 carcinogens
- Sudan dyes have been reported as contact allergens and sensitisers.

Regulations:

- Sudan dyes are not permitted colours in food regulations of many countries/agencies (e.g., UAE, EU, Australia, Canada, China, Hong Kong, ...).
- Their presence, at any level is not permitted in foods

Introduction:

- Sudan dyes are Synthetic chemical dyes of similar chemical structure.
- They are oil-soluble, aromatic compounds containing azo group (-N=N-)
- Sudan I,II,III and IV are red dyes that are used for colouring hydrocarbon solvents, oils, waxes, petrol, plastics and shoe & floor polishes.
- o Added to foods such as chilli powder to mimic/intensify their natural hues.

Characteristics	of Sudan	dyes studied

Sudan dye	CAS Number	CI No	Chem. Class	Molecular Formula	Molecular weight
Sudan I	842-07-09	12055	Azo	$C_{16}H_{12}N_2O$	248.28
Sudan II	3118-97-6	12140	Azo	$C_{18}H_{16}N_2O$	276.33
Sudan III	85-86-9	26100	Azo	$C_{22}H_{16}N_4O$	352.39
Sudan IV	85-83-6	26105	Azo	$C_{24}H_{20}N_4O$	380.44
Sudan Red 7B	6368-72-5	26050	Azo	$C_{24}H_{21}N_5$	379.46
Para red	6410-10-2	12070	Azo	$C_{16}H_{11}N_3O_3$	293.28

Characteristics of Sudan dyes studied					
Sudan dye	Melting Point (° C)	λmax (nm) (Toluene)	Chemical name		
Sudan I Sudan II Sudan III Sudan IV	131-133 156-158 199 (dec.) 199 (dec.)	476 (418) 493 (420) 507 (354) 520 (357)	1-phenylazo-2-naphthol 1-(2,4-Xylylazo)-2-naphthol 1-[4-(Phenylazo)phenylazo]-2-naphthol 1-[2-methyl-4-(2-methylphenylazo) phenylazo-2-naphthol		
10/3 / 1	7B130 (dec.)	535 (372)	N-ethyl-1-[[p-(phenylazo)phenyl]azo]- 2-naphthalenamine		
Para red	248-252	488 (328)	1-(4-Nitrophenylazo)-2-naphthol		

Chemical Structures of Sudan dyes

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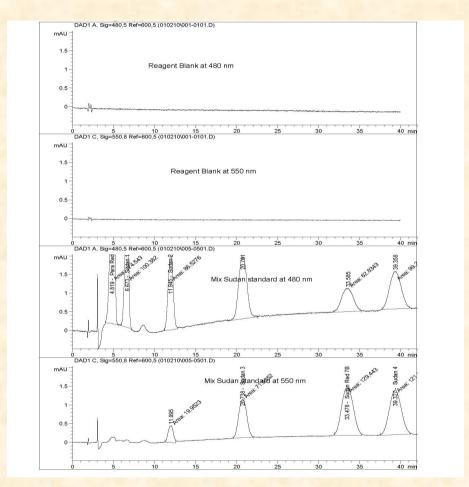
Sudan dyes – Analytical Methodology

- 1) Sudan dyes are extracted from sample using mixed solvent (Acetonitrile & Acetone)
- 2) Sudan dyes in the clear extract are separated by HPLC
 - a) Column: Lichrosorb 10 RP C-18 (4.6 mm i.d x 250 mm)
 - b) Mobile phase: 10 mM NH₄OAc in water (pH: 3.6) + Acetonitrile at 1.5 mL/min
- 3) Sudan dyes are detected by PDAD at 480 nm & 550 nm.

STANDARD CHEMICAI SOLUTIONS

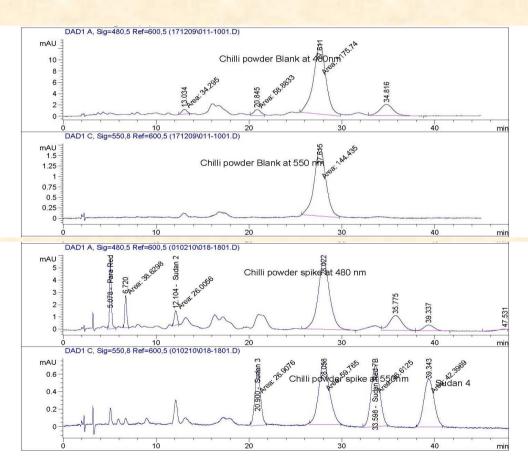
Standard Solution (2 mg/L)

Reagent Blank

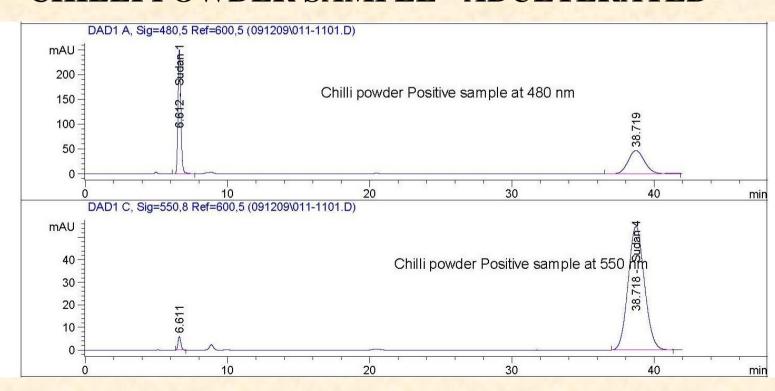


CHILLI POWDER SAMPLE

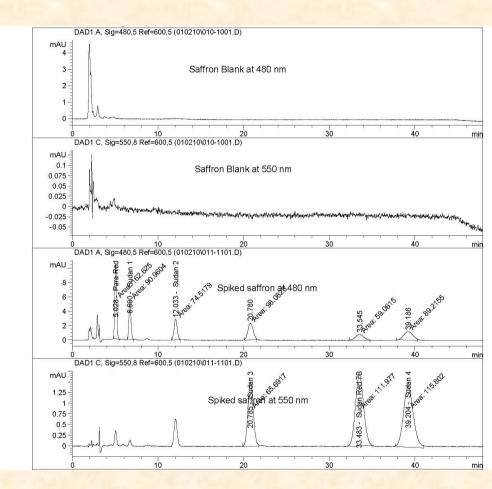
Pure Sample Spiked Sample 5 mg/kg)



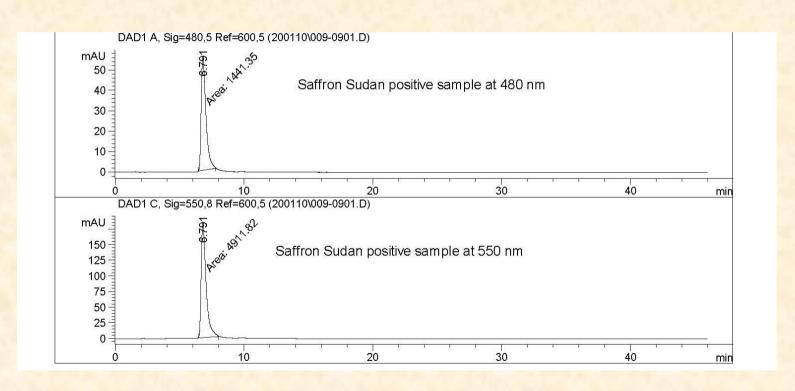
CHILLI POWDER SAMPLE - ADULTERATED



SAFFRON SAMPLE Spiked Sample Pure Sample (20 mg/kg)



SAFFRON SAMPLE - ADULTERATED

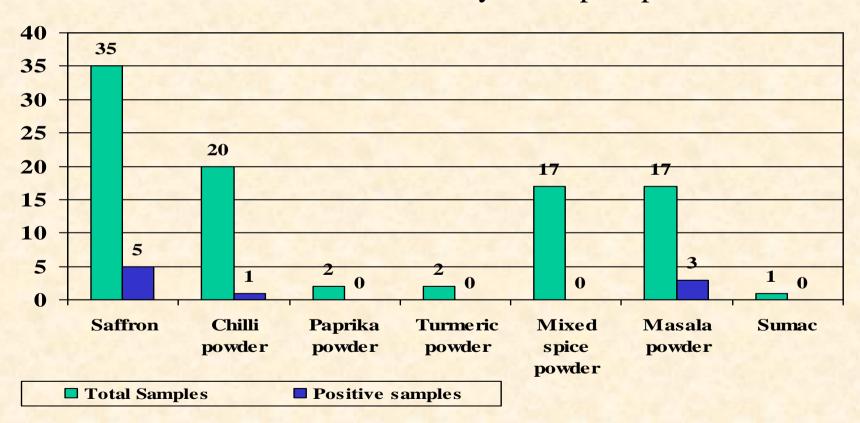


Sudan dyes – Method performance

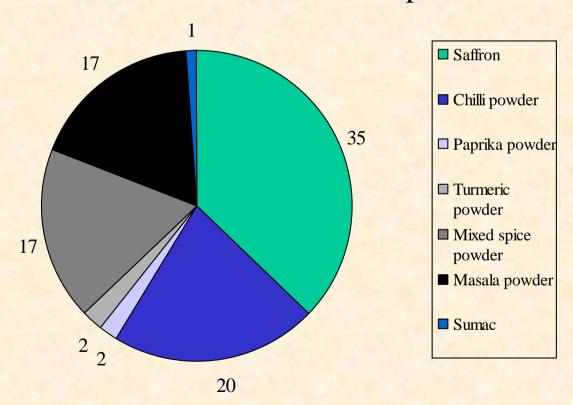
- 1) Inter day precision: % RSD 8
- 2) Limit of detection: 0.3 mg/kg of Sample
- 3) Linear range of Calibration graph: up to 20 mg/L with 20 µL injection volume
- 4) Recovery (for various matrices & in the spike level range of 1 to 20 mg/kg): 70 to 110 %
- 5) Chilli/Saffron matrix components did not give any peaks close to those of Sudan dyes.

S	Surveillance of Sudan Dyes in Spice products					
SI.	Name of Material	No. of	Positive Samples			
No.		Samples tested	Number	Dye	Level (mg/kg)	
1	Saffron	35	5	Sudan I	1.5 - 250	
2	2 Chilli powder	20	1	Sudan I	1599	
				Sudan IV	666	
3	Paprika powder	2	0	-		
4	Turmeric powder	2	0	-		
5	Mixed Spice powder	17	0	-		
6	Masala powder	17	3	Sudan I	1.5,3,1063	
7	Sumac	1	0		-	
	TOTAL	94	9			

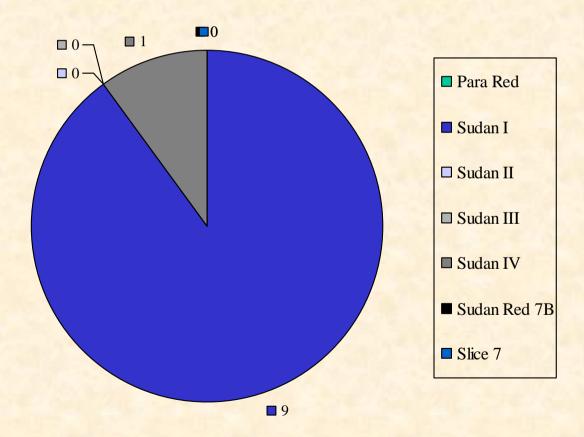
Surveillance of Sudan dyes in Spice products



Surveillance of Sudan dyes in Spice products
- Number of Samples



Sudan dyes in Spice products – Number of Positive samples



Conclusions

- 1) Method standardised at F&ELS is found fit to detect Sudan I IV, Para red & Sudan Red 7B at levels of current regulatory recommendation.
- 2) Chilli / Saffron matrix components did not interfere with analytes under consideration
- 3) Presence of Sudan dyes in some of the samples tested indicates the necessity of continuing this activity.

Working Group:

Dr. A. G. Krishnamacharyulu & Mrs. Vaidehi Garimella

THANK YOU

For your

Cooperation &

Patient listening

-Dr. Gopalakrishna