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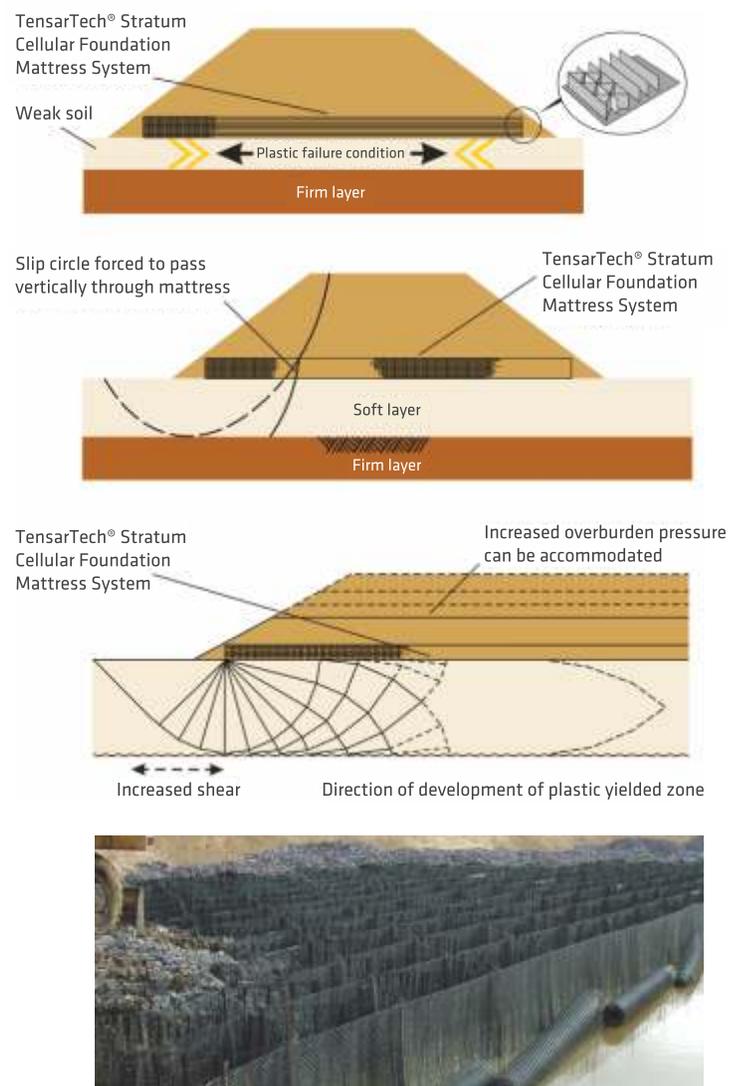
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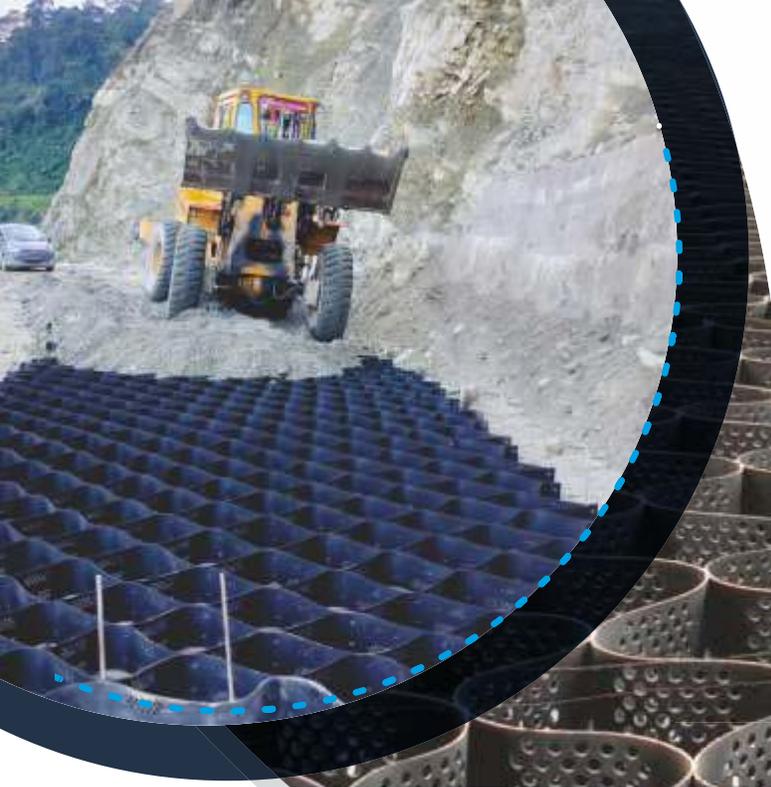
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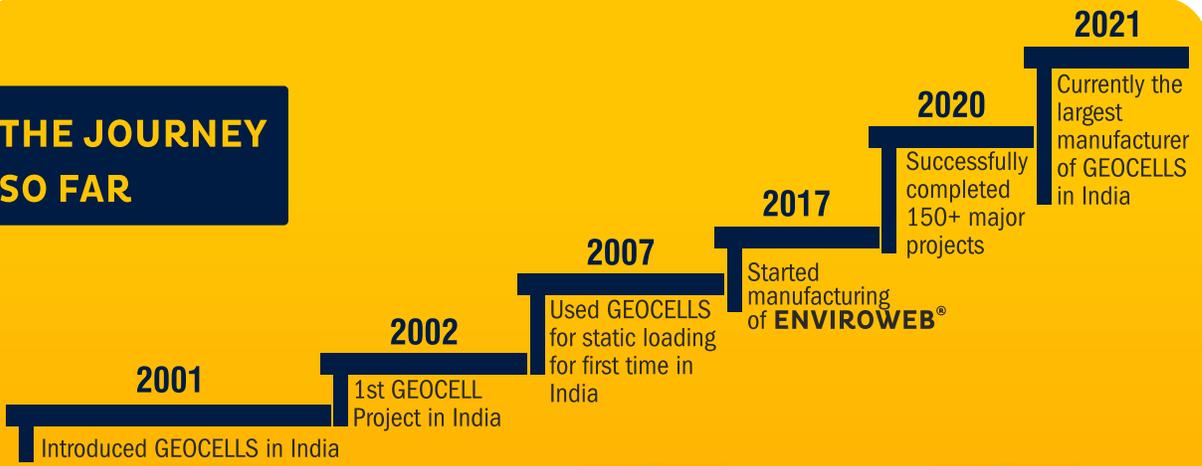


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EDITOR'S DESK

Dear Readers,
Greetings!!

Research with persistent and focused efforts lead to a positive result. Fostering research and providing a platform to publish quality research papers and related articles has been a continuous effort of BTRA Scan. In continuation to this effort, I am delighted to present to our readers the 4th issue of 50th Edition of BTRA SCAN. Slowly we are coming out of the pandemic and challenging period. We have to now focus fully on our progress and development.

This issue has papers from the different domains such as natural dye, effluent treatment, hexavalent chromium analysis and geotextile installation damage. Now we are open for authors from outside so researchers can send their original articles, case studies, research reviews or empirical contributions for publication in our journal.

The future is undoubtedly positive for the growth of industries. I feel we will have a great time ahead. Hope, we all are following the safety practices to keep away the pandemic from our life.

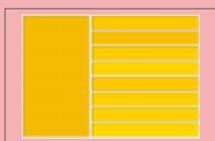
Our sincere thanks to all the reader and contributors for their support and interest.

T V Sreekumar, PhD
Director, BTRA

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Zero Liquid Discharge ETP – A Case Study (Part III)

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Abstract

The treatment steps like primary, secondary, and advanced filtration in Textile ETP are already discussed. After advanced filtration, the reject effluent management and the salt recovery are the important steps to achieve complete ZLD. Multiple effect evaporation, crystallization, Nanofiltration, and Agitated Thin Film Drying are the equipment used for this purpose. The changing TDS mass balance at each stage has been discussed. The experimental initiatives to use recovered Glauber salt and mixed salt are necessary and expected from dyers.

Keywords

Textile, ZLD, ETP, RO reject TDS mass balance, MEE, crystallizer, ATFD, salt recovery.

1.0 Introduction

In the part I&II of this article series, we have understood the characteristics of the influent and basic effluent treatment scheme, components of ZLD, and advance filtration techniques including nanofiltration and reverse osmosis technique to get ur water from the textile effluent. Here, as we are discussing a case study of 600KLD (600000 liters of the effluent/day) effluent from a yarn dyeing plant. The described treatment scheme is as per the case study. We have also studied the effect of these processes on the effluent and accordingly actual test results of inlet and outlet effluent characteristics were discussed stagewise. A complete treatment scheme to achieve Zero liquid discharge was shown with mass balance. Now in this last and III part, we will be discussing the multiple-effect evaporation, mass balance, salt recovery, Sludge generation, and disposal part so that complete ZLD is achieved

2.0 RO Reject characteristics and Nanofiltration output

For 600 KLD plant with 4 stages RO, the RO reject volume will be 51-54 KL with a TDS level of 67000 mg/lit.

Thus for RO reject quantity of 50 KL with TDS level 68000 ppm, the total salt content in the reject is approximately 3400 kg. In this 3400 kg salt, approximately 2550 kg will be common salt with other mono-valent salt and approximately 850 kg will be the Glauber salt.

When this RO reject effluent is further passed through nanofiltration then in the permeate common salt and other

monovalent salts will be allowed and Glauber salt and some quantity of mono-valent salt will be in the nano reject

The mass balance observed during nanofiltration is as given below in table 1.

Table 1 – The TDS mass balance during nanofiltration

Particulars or operations	
RO reject and feed to Nanofiltration	50 KL
Nano permeate quantity	25 KL
Nano permeate TDS level	68000 ppm
The common salt and other monovalent salt quantity in nano permeate	1700 kg
Nano filter reject quantity	25 KL
Glauber salt and other mono-valent salt quantity in Nano reject	1700 kg

This Nano reject quantity is taken for Multiple effect evaporation (MEE) for further product concentration

3.0 Salt recovery and its ZLD need

We know that two types of salts are used in the textile dyeing process i.e. Common salt and Glauber salt depending on the shade type, depth, and dyes criticality. Generally, this ratio is 3:1 to 4:1. Additionally, during the neutralization process, the salt is generated due to acid and base reaction, and thus

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the salt burden and hence toxicity is more in the textile effluent. Due to salt and other toxicity, the environmental issues being occurred are as below

3.1 The adverse impact of salt on land or soil

- The fertility of the soil is decreased and after a certain time it is useless for agriculture purpose
- Acidification of land increases
- The porosity of land reduces and the underground water level also decreases
- The crop taken from such land is contaminated

3.2 Adverse impact on a water body

- It is toxic to fish and reduces dissolved oxygen levels in the water
- Endocrine disruption of fish and thus reproductive system is hampered
- Increases algae growth and dead zones are developed in the sea

3.3 Concept and Need of ZLD

ZLD means Zero liquid discharge i.e. Zero discharge of wastewater from Industries. Zero Liquid Discharge (ZLD) is a treatment process designed to remove all the waste/pollution from water In other words while running any textile production activity, you will not discharge water (waste or pollutants) to the environment.

A ZLD system involves a range of advanced wastewater treatment technologies to recycle recovery and reuse the treated wastewater and thereby ensure no discharge of wastewater to the environment.

ZLD concept is not only treatment of wastewater, but it also talks about 3R i.e. Recover, reuse, and recycles. To make the above things successful and economical to the business module we should also focus on 4th R i.e. Reduce use at source. In short:- The focus of ZLD is to reduce wastewater economically and produce clean water that is suitable for reuse (e.g. irrigation, production), thereby saving money and being beneficial to the environment as well as business.

3.4 Economy Process sequence followed

The following process sequence is used to achieve complete ZLD, as shown in figure 1

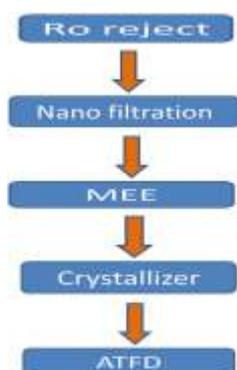


Figure 1 - Salt recovery process sequence

4.0 Multiple Effect Evaporation (MEE)

4.1 MEE parts and working:-

The evaporator consists of a large cylindrical body with a dome-shaped top and bottom. Inside, calandria are fitted.

Calandria consists of the number of vertical tubes (diameter 5-9 cms and length 3-7 meters).

When such evaporators are arranged in sequence it is called MEE. Multiple effect evaporators each is held at a lower pressure than the last. The vapor from 1st evaporator is a heating media for 2nd evaporator. Similarly, vapor from the 2nd evaporator serves as heating media for the 3rd evaporator and so on. Generally, 4 to 5 stages of MEE are used in the ZLD plant. The last evaporator is collected to cyclone separator (for gas separation). The steam economy increases as the stages are increased. For 4 stage MEE, to evaporate 4 liters of water, the steam required is 1 kg. For evaporation falling film-type evaporator is used. The typical diagram and its parts are shown in below figure 1.

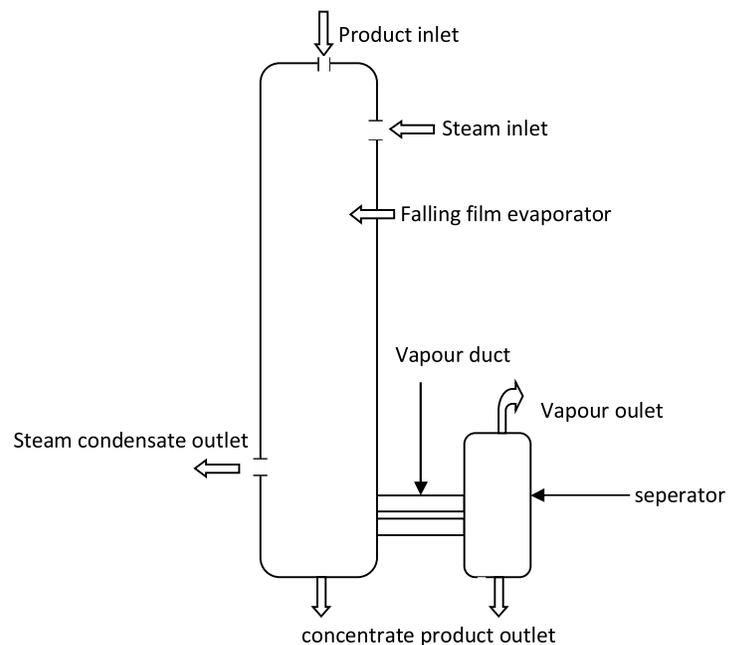


Figure 2- Evaporator machine parts [1]

The mass balance after MEE operation observed is as given in table 2

Table 2 - The TDS mass balance after MEE operation

Particulars or operations	
MEE feed	25 KL
Feed rate	5 KL/hour
MEE concentrate /product quantity	6 KL
MEE concentrate /product TDS level	280000 ppm
MEE condensate water from effluent with TDS level 100 ppm	19 KL

4.2 Preventive Maintenance of the MEE:-

To achieve desired machine operation efficiency and cleaning, the preventive maintenance schedule followed in the mill is as below in table 3

Table 3- MEE Preventive maintenance schedule

MULTIPLE EFFECT EVAPORATOR		
Maintenance Schedule		Agency
Daily	The specific gravity of effluent from last Evaporator/Calendria Steam pressure, Vacuum & Temperature calandria, Flow of effluent (to know chock-up of tubes)	Own team
Weekly	Flushing by water & chemical if needed	Own team
Yearly	Cleaning of tubes by motorized scrubber & high-pressure pumps (Jet pump)	Outside team

4.3 Cleaning of MEE

During the evaporation, the scale is formed inside the tube due to the deposition of insoluble salts/ hard scale (ex. sodium carbonate and bicarbonate). The higher the scaling lower is the efficiency. Hence the cleaning is required. The following figure 3 explains the sequential chemical cleaning method and steps used in industry.

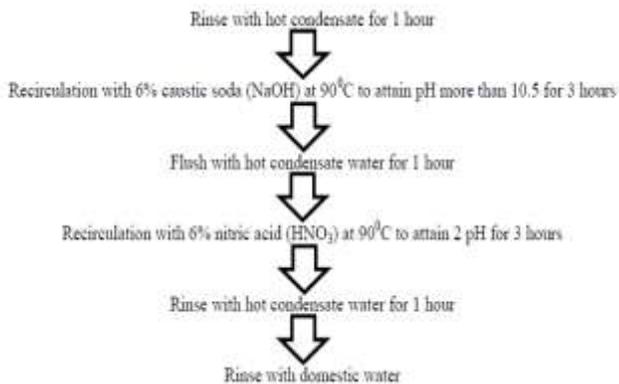


Figure 3:- Sequential chemical cleaning procedure of MEE [2]

5.0 Crystallizer

5.1 working of crystallizer

The MEE concentrate product is then fed to the Crystallizer unit. Crystallization refers to the formation of solids crystals from a supersaturated homogeneous solution

Crystallizers are used for recovering salts from wastewater (concentrated form) that can be reused in the process or sold in the market. Mainly two types of crystallization processes are used i.e 1) Concentration method (example Forced circulation crystallizer) and 2) Cooling (under vacuum or with a heat exchanger)

Vacuum cooling crystallization is mainly used for salts with a decreasing solubility at decreasing temperatures. By lowering the temperature, the salt crystallizes. This is held in suspension by air sucked in at the bottom of the crystallizer (air agitation) and is transported to the outlet

Economy process sequence:-

RO reject Nano filter (to separate common salt etc) MEE crystallizer ATFD

After Crystallizer, the produced Glauber salt is pure form, and mother liquor containing mixed salt is further transferred to ATFD for drying.

By flashing the solution (with sp gravity 1.2 – 1.25) in a vacuum, the solution temperature is reduced. This reduces the solubility of the Glauber salt and causes crystallization. The resulting vapor steam can be used for preheating. The crystallizer is working under vacuum 750 mmHg (1 kg/cm2) and 10 to 15°C temperature to recover Glauber salt(Sodium Sulphate), Temperature is maintained with the help of the chiller plant.

Output Glauber salt is with moisture about 50 %,which be dried with the help of ATFD type dryer or so that powder form Glauber salt can be achieved with 10 % moisture level. The % purity of Glauber salt obtained is >90%

The mass balance during the crystallization process is given in table 4

Table 4 - The TDS mass balance in crystallization

Particulars or operations	
Crystallizer feed	6 KL
Glauber salt quantity	2 KL
Obtained Glaber salt concentrated product purity	50%
Glaber salt weight on a dry basis	1000 kg
Mother liquor quantity	4 KL
Mother liquor TDS level	175000 ppm

5.2 Preventive maintenance of crystallizer:-

The preventive maintenance schedule for crystallizer followed is given in table 5

Table 5- Crystallizer : Preventive maintenance schedule

Crystallizer and pusher		
Maintenance Schedule		Agency
Daily	The temperature of the vessel.	Own team
Weekly	Vacuum	Own team
Monthly	Cleaning & Flushing	Own Team
Yearly	Cleaning by chemicals Overhauling	Outside team

6.0 Reuse of recovered salt

6.1 Common salt

It is recovered from nanofiltration and is in the highly concentrated form (TDS level generally is 50-70000 ppm). It can be used directly in the form of liquid by considering the % purity.

6.2 Glauber salt

It is recovered in the form of crystals (moisture content is about 45-50 %) It can be reused for dyeing purposes by considering its moisture content. Even if recovered Glauber salt with a moisture content level of 50 % can be used in the dyeing as a brine solution. Only one should look into bath volume and salt solution to be added. Recovered brine quantity to be added < dyeing bath volume. So, it is proffered to use the recovered brine solution as it is instead of dry powder. This saves the drying cost of salt recovered. For your ref, please note that almost all the shades can be dyed with this recovered salt. The only dyer has to take experimental based initiative for this.

7.0 Final drying by ATFD

7.1 ATFD machine parts and working

The mother liquor coming from the crystallizer and pusher system is taken to the ATFD (agitated thin film dryer) for further concentration from 20 % to 90% TDS level so that almost complete water evaporation is achieved ATFD-agitated thin film dryer – is used for evaporation of water to make concentrated liquid to dry powder form-continuous process.

ATFD consists of a cylindrical vertical body with a heating jacket and rotor inside the shell- equipped with row and pendulum blades all over the length of the dryer

The hinged blades spread the wet, feed liquid in a thin film over the heating wall

The turbulence increases as the product pass through a clearance before entering calming zone situated behind the blades as the heat will transfer from jacket to wall under the smooth agitation

Water evaporates and liquid converts to slurry to cake to dry powder

The vapors move upward and pass through the cyclone separator at the top. These vapors are condensed to form condensate water

The liquid is fed from Top. The ATFD Temp is approx. 150 deg C. The mother liquor from crystallizer is converted to powder form & is continuously scrapped with help of blades in ATFD and dry salt with moisture level 8-10 % is produced. This salt is called mixed salt

The following figure no.4 shows the different parts of the ATFD machine.

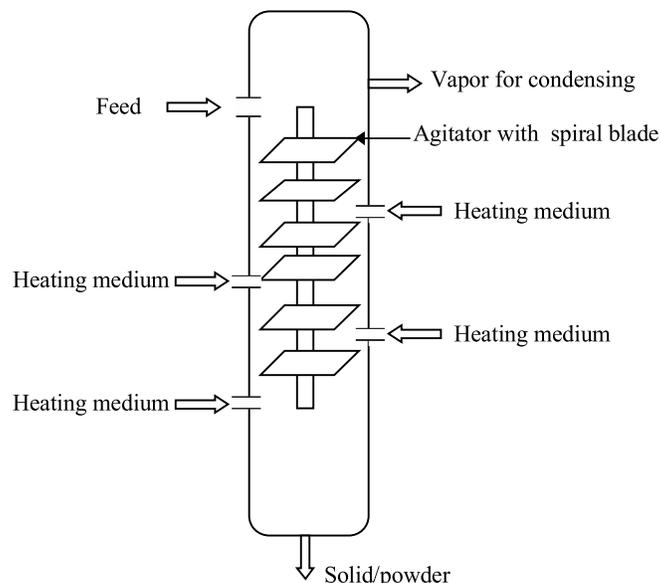


Figure no.4 - Agitated thin film dryer (ATFD) machine parts [3]

7.2 Preventive machine maintenance of ATFD:-

The preventive maintenance schedule for the ATFD machine followed is given in table 6

Table 6- ATFD machine Preventive maintenance schedule

ATFD		
Maintenance Schedule		Agency
Daily	Steam Pressure and temperature	Own team
Weekly	Cleaning & Flushing	Own team
Monthly	Cleaning & Flushing	Own Team
Yearly	Over-hauling	Outside team

8.0 Mixed salt Management

As of today, ATFD mixed salt is not used for dyeing purposes in wide scope. Generally, it is being discarded to waste management system i.e. Common Hazardous Waste Treatment, Storage and Disposal Facility (CHWTSDF) in your state /city/area. For example in the Mumbai zone, it is Mumbai waste management ltd is the agency working for this. But, a negative costing commercial is involved in dispatching such mixed salt to CHWTSDF. To save this cost dyer may take in experimental initiatives to use such salt for non-critical shades like black and navy etc.

9.0 Do's and Dont's in ETPP

- Use safety shoes or boots with non-slip soles
- Wear personal protective equipment and chemical resistant clothing to avoid exposure of skin or eyes to corrosive and/or polluted solids, liquids, gases, or vapors
- Do not mix chemicals without the supervision of a qualified chemist or safety professional.

- d. Obey all safety instructions regarding the storage, transport, handling, or pouring of chemicals. – as per MSDS
- e. Check electrical equipment for safety before use; verify that all-electric cables are properly insulated; take faulty or suspect electrical equipment to a qualified electricity technician for testing and repair
- f. Wear safety goggles in all cases where the eyes may be exposed to dust, flying particles, or splashes of harmful liquids
- g. Wear a respirator, or gas mask, when exposed to harmful aerosols, dust, vapors, or toxic gases
- h. Take extreme care when handling highly corrosive agents such as liquid or gaseous chlorine, concentrated acids or alkalis, etc
- i. Obey all safety instructions concerning entry into confined spaces, e.g., check atmosphere for oxygen or for poisonous gases, use respiratory protection equipment if needed, have a co-worker stand guard in case of need for help, etc.
- j. Do not smoke, eat or drink in areas where chemical or biological contamination may be expected
- k. Use non-latex gloves if the sensitivity to latex has been diagnosed
- l. All operators should undergo periodic examinations by an occupational physician to reveal early symptoms of possible chronic effects or allergies

Conclusion

ZLD concept is not only treatment of wastewater, but it also talks about 3R i.e. Recover, reuse, and recycle. This is not only applicable to water but also the salt and other toxicity in the effluent. To make the above things successful and economical to the business module we should also focus on 4th R i.e. Reduce water and chemicals used at the source. In short:- The focus of ZLD is to reduce wastewater economically and produce clean water that is suitable for reuse (e.g. irrigation, production), thereby saving money and being beneficial to the environment as well as business.

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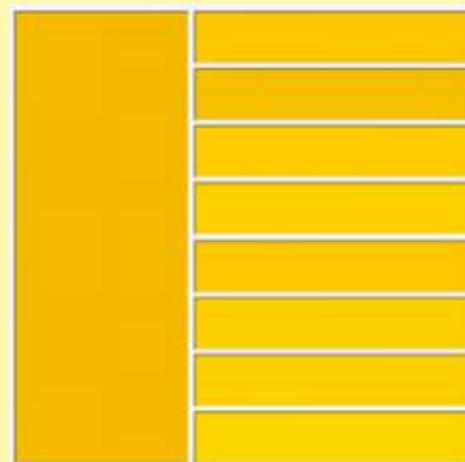
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Improved Fastnesses through Modified Turmeric Dyeing using Rare Earth Salts As Mordants

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Abstract

Turmeric dyeing of silk fabric with Rare earth salts as mordants has been attempted to improve the wash and lightfastness of dyed fabrics. Two different mordanting methods were adapted- pre and post mordanting with 6 different rare earth salts, namely Cerium nitrate, Lanthanum carbonate, Yttrium oxide (series-1), Cerous sulphate, Lanthanum chloride and Yttrium chloride (series-2). A conventional mordant- alum was also used for comparison. The silk fabric (RFD) was used for dyeing with the same concentration of Turmeric dye to assess the effect of Rare earth salts on the colour strength of the dyed fabric and fastness properties.

Keywords

Turmeric dye, Cerium, Lanthanum and Yttrium salts mediated natural dyeing, improved fastnesses

1.0 Introduction:

Turmeric dyeing has always posed a challenge to dyers. The colour adherence and retention of the dyed fabric is a major concern. It is known to us that reflectance is inversely proportional to absorbance, so when absorbance is less, reflectance is more and vice versa. Shade percentage often influences the reflectance percentage of the dyed fabrics. The colour strength of a dye is a measure of its ability to impart colour to the dyed material. This property is characterized by the absorption in the visible region of the spectrum and can be expressed as a colour strength value. Colour strength (K/S) is the most important parameter to test the quality measurement of a sample in terms of the depth of the colour dyed fabric. The natural dye extracted from Turmeric was used for dyeing cotton at different dyeing conditions by Umbreen et al [1]. The fastness properties of the dyeing showed to have good saturation and rubbing fastness, but poor washing and light fastness properties on cotton without any mordant. However, when the dyeing was done with mordants, washing and light fastness properties improved while rubbing fastness exhibited deterioration. Adeel et al [2] observed that UV radiation enhances the colour strength of dyed fabric which was irradiated using a low concentration of dye. The colour fastness properties of

dyed fabrics which were pre-irradiated cotton fabric and dyed by using pre-irradiated Turmeric powder was found to be good. Shukla and Vankar [3] attempted to activate the Curcumin molecule by complexation with chitosan. The binding took place at intrinsic pH (7–8) very effectively without any surfactant or enzyme. Dyeing with this composite showed excellent wash and light fastnesses as compared to Curcuma dye. Improved fastnesses and wash cycle sustenance were observed.

This research aims to find solutions to three major problems commonly faced with Turmeric dye. We were focussing on the following points. They are -1. Are there simpler ways to make Turmeric dye non-fugitive? 2. How can dye depths be increased for dyed fabric? 3. How can we enhance the wash and light fastnesses of Turmeric dyed fabric?

2.0 Materials and Methods

2.1 Materials

Turmeric powder was procured from the local market (MDH), Rare earth (RE) salts, namely Cerium nitrate, Lanthanum carbonate, Yttrium oxide, Cerous sulphate, Lanthanum chloride and Yttrium chloride were procured from Indian Rare Earths Ltd. (IREL), Head Office, Mumbai. Ezee soap solution was procured from the local market, it was used for washing the dyed silk fabrics. Citric acid was procured from Merck. Silk fabric was purchased from Sanjay Shah and Associate Company.

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2.2 Methods

2.2.1 Mordanting

The process of mordanting was carried out by adopting the pre-mordanting method using 1 % of the mordant solution and maintaining a 1:30 material-to-liquor ratio. The process of pre-mordanting was carried at 50–55 °C with gentle stirring and continued for 45 minutes, for the mordants-Cerium nitrate (CN), Lanthanum carbonate (LC), Yttrium oxide (YO) the process of pre-mordanting was with 1:1 solution made with citric acid (CA) and RE salt (Series-1) and the process for Cerous sulphate (CS), Lanthanum chloride (LC) and Yttrium chloride (YC) using only RE salts (Series-2). It was carried out similarly. For post-mordanting, the Turmeric dyed swatches were prepared and then the mordanting with RE salts: CA (Series-1) and RE salt (Series-2) were carried out. Time and temperature of mordanting were kept the same as pre mordanting method.

2.2.2 Extraction of Turmeric dye

The dye was prepared by taking 5 % of the Turmeric powder concerning on weight of fabric (OWF) of the silk swatches. The powder was heated in deionized water for 30 minutes and then filtered. The filtrate was used directly for dyeing the silk fabric.

2.2.3 Dyeing of the silk fabrics

The process of the dyeing was carried out in a water bath by maintaining the material-to-liquor ratio of 1:30 for silk fabric. All the test fabrics were dipped in 200 ml of dyeing solution of 5 % dye concerning on weight of fabric (OWF) at 50-55°C temperature. The material was then removed and washed with 1 % of mild detergent (Ezee) and water 2–3 times followed by drip-drying operations at room temperature.

2.2.4 Measurement of reflectance (%) & colour strength (K/S)

Reflectance (%) of the dyed fabric samples were measured by using a Premier Colour scan spectrophotometer. As the strength of any dye is related to its absorption property, so by using Kubelka–Munk formula the following relation between reflectance and absorbance can be derived:

$$K/S = \left\{ \frac{(1-R)^2}{2R} \right\}$$

Where R is the reflectance, K is absorbance and S is the scattering. By using the above equation colour strength of different Turmeric dyed samples were measured.

2.2.5 Fastness properties of the dyed silk fabric

The standard procedure ISO: 105 C: 10 was followed to measure the washing fastness. Launder-o-meter was used to washing the samples. fathometer with xenon arc lamp was used to test the lightfastness with the standard procedure ISO: 105- B02: 2014.

3.0 Results and Discussion

3.1 CIE Lab and K/S values of Turmeric dyeing samples

The Rare earth mordants of series-1 were used for pre and post mordanting and dyed swatches of silk using turmeric as the natural dye source. The results are shown in Table 1. Table 1 shows the CIELab values, K/S values and ΔE. The best dyeing results were obtained from pre mordanted Cerium nitrate/citric acid sample.

{Series 1: Standard – Control, Batch-1 Alum pre mordanted, Batch-2 Alum post mordanted, Batch-3 Cerium nitrate/Citric acid pre mordanted, Batch-4 Cerium nitrate/Citric acid post mordanted, Batch-5 Lanthanum carbonate/Citric acid pre mordanted, Batch-6 Lanthanum carbonate/Citric acid post mordanted, Batch-7 Yttrium oxide/Citric acid pre mordanted and Batch-8 Yttrium oxide/Citric acid post mordanted}

Table-1 CIELab and K/S values of Turmeric dyeing samples (Rare earth pre and post mordanted) Series-1

S. No	Name	K/S	L*	a*	b*	C*	H*	ΔE*	Remark
1	Standard	95.36	76.94	7.16	85.46	85.75	85.17	--	
2	Batch-1	98.32	80.61	5.57	93.28	93.45	86.17	8.79	
3	Batch-2	104.35	78.24	4.96	91.01	91.15	86.84	6.12	
4	Batch-3	120.37	82.99	6.21	101.75	101.94	86.47	17.41	Best
5	Batch-4	95.37	83.54	1.20	96.13	96.15	89.25	13.89	
6	Batch-5	120.10	81.16	8.50	99.38	99.74	85.07	14.61	
7	Batch-6	82.41	83.11	1.84	93.17	93.19	88.83	11.22	
8	Batch-7	95.39	83.43	2.60	96.53	96.57	88.41	13.62	
9	Batch-8	74.09	84.48	-0.85	93.46	93.47	90.56	13.61	

The Rare earth mordants of series-2 were used for pre and post mordanting and for dyed swatches of silk using Turmeric as the natural dye source. The results are shown in Table 2. Table 2 shows the CIELab values, K/S values and ΔE . The best dyeing results were obtained from pre mordanted Cerous sulphate sample.

However, grades of the mordanted samples recorded showed better results than that of the unmordanted samples. In most of the RE, salts pre-mordanting was found to be better in terms of K/S values, however, in the case of alum, it was

posted mordanting which gave better results in the case of series-1 and 2 of RE salts. Only Yttrium chloride of series-2 showed better results in post mordanting.

{Series 2: Standard – Control, Batch-1 Alum mordanted, Batch-2 Alum post mordanted, Batch-3 Cerous sulphate pre mordanted, Batch-4 Cerous sulphate Post mordanted, Batch-5 Lanthanum chloride pre mordanted, Batch-6 Lanthanum chloride post mordanted, Batch-7 Yttrium chloride pre mordanted and Batch-8 Yttrium chloride post mordanted}

Table-2 CIELab and K/S values of Turmeric dyeing samples (Rare earth pre and post mordanted) Series-2

S. No	Name	K/S	L*	a*	b*	C*	H*	ΔE^*	Remark
1	Standard	100.06	78.87	5.67	89.61	89.79	86.34	--	
2	Batch-1	82.62	80.48	3.72	88.82	88.90	87.56	2.65	
3	Batch-2	104.34	78.23	4.96	91.01	91.15	86.84	1.69	
4	Batch-3	119.60	81.70	5.68	99.15	99.33	86.58	9.95	Best
5	Batch-4	102.92	78.56	1.68	89.96	89.97	88.89	4.01	
6	Batch-5	114.37	76.69	8.50	99.38	99.74	85.07	7.08	
7	Batch-6	97.50	75.58	10.07	85.00	85.60	83.20	7.17	
8	Batch-7	91.08	78.77	6.96	87.32	87.60	85.40	2.63	
9	Batch-8	100.37	75.54	9.43	84.97	85.49	83.62	6.84	

3.2 Fastness properties of the Dyed silk fabrics

Table-3 Fastness properties of silk swatches

Turmeric dyed samples	Wash Fastness		Light Fastness	
	Pre mordanting	Post mordanting	Pre mordanting	Post mordanting
Control	3	3	1	1
Alum	4	4	1	1
Cerium nitrate	4-5	4-5	1	2
Cerous sulphate	4-5	5	1	2
Lanthanum carbonate	4-5	4-5	1	1
Lanthanum chloride	3-4	3-4	1	1
Yttrium oxide	4-5	5	1	2
Yttrium chloride	3-4	3-4	1	1

The colour fastness is determined and shown in table -3. The retention of the dye by the fabrics when exposed to different tests recorded yield the grades, indicating whether or not there has been efficient retention. The silk fabrics dyed with Turmeric dye recorded fair, fair to good, grades in the washing tests shown in table 3. Lightfastness recorded also showed improvement in the case of postmordanted cerium salts (nitrate and sulphate), as well as post mordanted yttrium oxide silk swatches. The overall retention of the dye in combination with the different RE salt mordants showed the least fading and bleeding. However, the unmordanted dyed samples also showed satisfactory K/S, but the fastness properties were poor. The washing fastness of the dyed silk swatches with RE salts showed good influence on the rate of

the diffusion of the dye and the state of the dye which has penetrated and its adherence. The Colour strength (K/S) of the dyed fabric has been found to influence fastness value. In this research, a relation was found between Colour Strength (K/S) and dye adherence.

4.0 Conclusion:

The three objectives were fulfilled as the dyed silk swatches with RE salts showed good dye adherence and washing fastness. Lightfastness improved marginally. Work is in progress to make it better. Thus it can be concluded that using RE salts in conjunction with Turmeric dye seems to be an industrially viable proposition.

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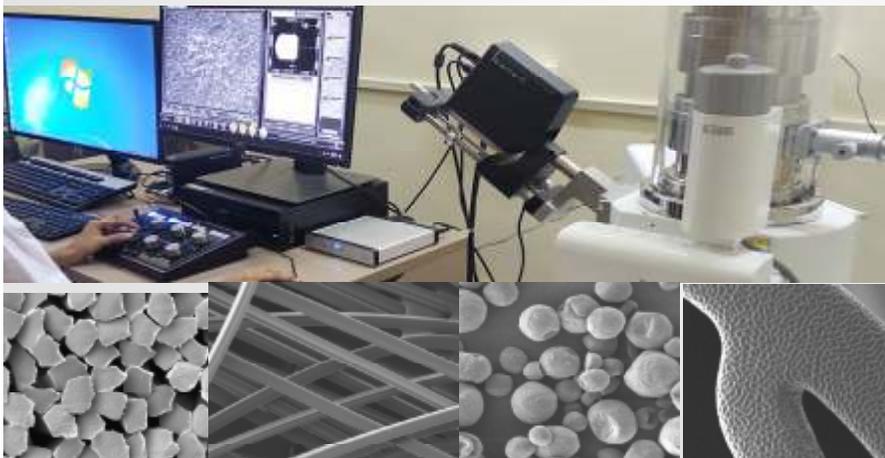
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Comprehensive Overview of Installation Damage Test on Geo-textiles

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Abstract

Nowadays soil reinforcement is necessary for geotechnical engineering and geo-textiles are being used as reinforcement since it is easy to install, long-lasting, and economic. Installation damage can be expected to modify the mechanical properties of geosynthetics. The study and investigation into the long-term performance, design life, and survivability of geo-textiles, especially due to installation damage are necessary. During installation, spreading, and compaction of backfill materials, geotextiles may encounter severe stresses which can be higher than they will experience in-service. This paper contributes to a better understanding of how installation damage affects the design life of geo-textiles and the importance of installation damage. Apart from these, steps taken to minimize the installation damage and test procedure to conduct the installation damage test are also discussed. Several authors have demonstrated that installation damage can be minimized to a larger extent by following standard installation procedures. The coarse backfill causes severe installation damage to the geotextiles so care should be taken accordingly to reduce the damage. The installation damage test, provide the reduction factor value of the geo-textile compared to the control specimen before the actual installation, which can be considered to evaluate the life of the design. Therefore, it is significant to perform the installation damage test of the geo-textiles.

Keywords

Geo-textiles, installation damage, reduction factor, tensile strength, design life

1. Introduction

Nowadays geo-textiles have become a part of road construction and soil stabilization techniques. The durability of the geo-textiles is one of the important parameters that need to be evaluated in design life. It depends on two characteristics namely degradation and resistance; degradation is caused mainly due to oxidation, ultra-violet radiation, hydrolysis, and chemical and biological agents(Hufenus et al. 2005). Resistance of geo-textiles towards installation damage, creep, stress relaxation, abrasion, and compressive creep. Among these, installation damage is a short-term effect that reduces the maximum tensile strength but does not affect the long-term properties such as creep and aging by hydrolysis, oxidation, and/or abrasion. The design period of the geo-textiles depends on the reduction factor. Hence, the durability of geo-textiles is assessed by the short-term accelerated tests under extreme conditions experienced in service(Cho et al. 2006). To establish the validity of these tests it is essential to compare their predictions with tests made on material from the site. Generally, the short-term properties after a certain period in

the service specimen are compared with the same properties determined on the control specimen. This will give the installation damage factor of the geo-textiles. If the installation damage factor for a particular geotextile is less, certainly, the short-term strength requirement will also be less.

It is a well-known fact that installation damage is inevitable for the geo-textiles and should be avoided (Fox et al. 1998). The installation of the geo-textiles comprises viz. removing soil, surface preparation, handling and laying geotextile, carrying, placing, spreading, and compacting the backfill material over the geotextile(Sardehaei et al. 2019). Consequently, it is impossible to avoid the installation damage of the geo-textiles but it is possible to minimize the damage by following adequate construction techniques; such as cleaning and clearing the sharp and roots from the surface where geotextiles are placed, wrinkles must be removed from the geo-textiles before laying, the backfill should fall from a minimum height of 0.15 m over the geo-textiles(Corbella and Stretch 2012; Vieira et al. 2015). It must be noted that construction equipment should be placed only on the backfilled geo-textiles. In fact, during the installation process, geotextiles may encounter more stresses

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than during their service life, with the appearance of cuts, frays, and general abrasion. The installation of geo-textiles in the coarser material causes severe damage to the hydraulic and mechanical properties

1.1 Overview of installation damage of geo-textiles

Generally, the design of geotextile incorporated soil structures takes the following two factors into consideration namely: the maximum strain in the geo-textile throughout the design period, which is considered as serviceability, and the minimum strength of the geotextile that can take to rupture, which is known as the ultimate limit state. These factors depend on time and are degraded by the environmental conditions to which the geo-textile is exposed. The design period (Td) for the geotextile soil structures is typically 50 to 100 years(Bathurst et al. 2011). However, this period is too long for direct measurements and to be made in advance of construction. Therefore, reduction factors are evaluated by extrapolation of short-term data, where necessary tests are conducted at elevated temperatures and loads to accelerate the processes of degradation(Miyata et al. 2015). The degradation strength of geo-textiles is time bound and is classified accordingly into three modes viz., immediate reduction, gradual, and no reduction. The immediate reduction is due to installation damage, which decreases the tensile strength(Lim et al. 2013).

The installation damage is the primary reduction factor applied in the design life of the geo-textiles. Koerner and Koerner (1990)examined 75 different geosynthetics textiles and stated the retained tensile strength after installation and excavation. The results revealed that coarse, irregular, and frozen subgrades, poorly graded cover soil with large particles, small lift thicknesses, and heavy construction equipment created severe damage. Furthermore, Allen and Bathurst (1994) summarized the results of tensile load-strain tests performed on different geosynthetic reinforcement products in site-damaged and undamaged conditions. They observed a greater loss of modulus for nonwoven geotextiles compared with woven geo-textiles, owing to the thinner fibers used in the nonwoven geo-textiles. Greenwood and Brady(1992) showed that the damage factor due to installation damage and the frequency of damage increased when increasing the backfill grain size and number of passes. Most researchers emphasize that the level of damage depends directly on the weight, type, and a number of passes of the compaction equipment. The installation damage will destroy geotextile's hydraulic efficiency, that is the ability to allow free passage of water through rock armor whilst retaining and protecting the soil beneath from washing away from tidal currents and wave actions. Geosynthetic clay liners (GCL) are different types of geosynthetics and are prone to damage through installation. Depending on the product type and hydration conditions, such stresses may damage the carrier geosynthetics, damage the reinforcement, or cause bentonite migration and consequent reductions in local mass per unit area of the product. Primarily based on the field observations during construction, ASTM D 6102 is the

most complete guideline for GCL installation currently available. However, there is a need for controlled field studies to substantiate and strengthen the recommendations of ASTM D 6102. Furthermore, neither ASTM D 6102 nor ASTM D 5818 provide details regarding field and laboratory procedures needed to conduct a controlled field test of GCL installation damage. Laboratory studies have investigated GCL bentonite migration under concentrated loads and areal load a controlled field study of GCL installation damage has not been reported. Watts and Brady (1990) developed a simple procedure with sufficient reproducibility for simulating installation damage on site. They showed that the tensile strength and the elongation at break were both substantially reduced by damage during installation, but Young's modulus was largely unaffected. Based on the literature results of installation damage for a wide range of geosynthetic reinforcements, the Federal Highway Administration (FHWA) has proposed the installation damage reduction factors according to Table 1. Apart from this, several researchers stated that creep strains are not affected by installation damage, unless the damage is severe, or unless the load level applied is very near the creep limit of the undamaged material (Allen and Bathurst 1994; Bathurst et al. 2011).

Table. 1 Installation damage factor as per FHWA recommendations

Geosynthetic type	Type 1 backfill size <102 mm D ₅₀ ~ 30 mm	Type 2 backfill size <20 mm D ₅₀ ~ 0.7 mm
Stretched biaxial PP grids	1.20–1.45	1.10–1.20
Stretched HDPE grids	1.20–1.45	1.10–1.20
PP slit tape woven	1.60–3.00	1.10–2.00
PVC coated PET grids	1.30–1.85	1.10–1.30
Acrylic coated PET grids	1.30–2.05	1.20–1.40
PP and PET woven	1.40–2.20	1.10–1.40
PP and PET nonwovens	1.40–2.50	1.10–1.40

2. Installation damage factor

The Geosynthetic Research Institute (GRI) Standard Test Method GG4 has given a method to determine the allowable strength and the long-term design strength of geo-textiles are calculated from Eq. 1 and 2 respectively, which is done by taking into consideration of the ultimate strength and the total Factor of Safety (FS) in the geo-textiles.

$$T_a = T_u \left[\frac{1}{RF_{id} + RF_{cr} + RF_{cd} + RF_{bd}} \right]$$

$$T_d = T_d \left[\frac{1}{RF_{id} + RF_{cr} + RF_{cd} + RF_{bd}} \right]$$

Whereas: Ta = allowable strength (N.m-1); Tu = ultimate strength (N.m-1), Td = long-term design strength (N.m-1),RFid = installation damage factor, RFcr = creep

deformation factor, RFcd = chemical degradation factor, RFbd = biological degradation factor.

In other words, the installation damage factor is an allowance given for the geo-textile to damage at site conditions during the installation process. It is defined as the ratio of the mean tensile strength of the undamaged material to the mean tensile strength of the damaged material. Pinho-Lopes et al. (2016) stated that the reduction factor for installation damage obtained from tensile tests overestimated the effects of the installation conditions on the soil–geotextile interface.

2.1 Methods to avoid installation damage.

To avoid installation damage, the following precautions should be taken(Black et al. 1999; Carlos et al. 2015):

1. The geo-textiles must be installed by the installation guidelines provided by the manufacturer or as directed by the Engineer.
2. The geotextiles should be provisionally fixed in place with pins, sandbags, or staples as per the fill properties, fill placement procedures or weather conditions.
3. To reduce the damage to the low strength geotextile a cushion layer of sand should be placed over it.
4. It must be ensured that geo-textiles are not prone to sunlight for more than the maximum duration permitted in the approved installation procedures. If the manufacture does not provide any specific guidelines, then the geo-textiles must be covered within a day of installation.
5. Care should be taken if the backfill of the soil is coarser and the backfill should fall from a height less than 1.5 m.

3. Installation damage test setup

The installation test is carried out as per the ASTM D5818, ISO 13437, and UK code of practice for geo-textiles. Primarily, the initial condition of geo-textile is identified such as identification and description of the structure, geo-textile environment, characteristics, and testing standard references. As per the standard specifications the installation damage test is carried out in three stages viz., 1. Installation of geo-textiles specimen, 2. extracting the specimen from the soil, and 3. testing on the extracted specimen in the laboratory(Jeon et al. 2010). The BTRA is planning to create the site for the installation of the specimens as per the specifications and then the testing of specimens extracted to evaluate the damage factor of the geo-textiles.

3.1 Installation and retrievals of the specimen.

To avoid the faulty test results ASTM D5818 and ISO 13437 have provided certain guidelines to follow: the number of samples is determined by the dimensions of the structure, the physical and chemical variations in the environment in which the geo-textiles is installed, and the repercussions that a failure of the geo-textiles function would cause. On the other hand, the number of retrievals depends on the design life. The installation damage specimen is taken after the test and the control specimen is taken from the material before installation and it should be as close as possible to the

material used in the installation damage specimen. Note: the dimensions of the installation damage specimen and control specimen are identical and should have an equal number of scheduled retrievals.

3.2 Installation damage test procedure

To initiate the testing, the subgrade was prepared and compacted using the vibratory compactor. Five steel plates are taken and the dimension of each plate is as follows 2m in length and 1.5m wide. The steel plates are equipped with steel chains and placed over the subgrade(Nikbakht et al. 2006). A layer of 200 mm thick soil/aggregates was then placed over the steel plates and then well compacted using the 10ton vibratory compactor. Five samples of geogrids, each of dimension 2m x 1.5m are placed over the compacted soil exactly where the steel plates were placed(Diederich 2000). The geogrids are placed in a way such that the machine direction of the geogrids is perpendicular to the direction of the compaction which represents the actual practice carried out in a reinforced soil structure. To complete the installation, a layer of 200 mm thick soil/aggregates was placed over the geogrids and compacted with the vibratory compactor. The compaction is done using a tandem steel-wheeled roller with vibration capability. Ten full roller passes are allowed to simulate the effective compaction as per the site conditions.

3.3 Exhuming the samples

After the completion of the testing, the samples shall be exhumed within 48 hours after the installation. To collect the exhumed samples, the soil/aggregate layer over the geogrids was carefully removed by lifting the steel plate to 45° with the help of lifting chains attached to the steel plates. Particular care is taken that there is no further damage to the geogrids while extraction. Exhumed samples shall be handled and stored in such a manner to eliminate, or minimize, further damage or degradation by exposure to ultraviolet light.

3.4 Gradation of the backfill material

As per ASTM D5818 specifications geo-textiles are exposed to the soil/aggregates of three different gradations. Soil types used in the tests are sandy gravel (BBA-1), gravelly sand (BBA-2), and silty sand (BBA-3) and are presented in Table.2 (Hufenus et al. 2005; Atmatzidis et al. 2009; Bathurst et al. 2011).

Table 2. Soil aggregate gradation for installation damage test

Installation DamageSoils / Aggregate gradation			
Sieve Size (mm)	Percentage Finer (%)		
	BBA-1 (Sandy Gravel)	BBA-2 (Gravelly Sand)	BBA-3 (Silty Sand)
75	100	100	100
65	99.60	100	100
37.5	85.78	100	100
26.5	-	100	100
19	69.48	100	100

Installation Damage Soils / Aggregate gradation			
Sieve Size (mm)	Percentage Finer (%)		
	BBA-1 (Sandy Gravel)	BBA-2 (Gravelly Sand)	BBA-3 (Silty Sand)
9.50	52.32	99.6	100
4.75	38.74	93.7	96.1
1.70	-	65.5	74.5
0.6	13.23	21.3	40.6
0.425	-	14	32.4
0.3	11.05	6.5	21
0.15	2	0.9	4.1
0.075	0	0.4	1.1
USCS Classification	GP	SP	SM
	Poorly graded gravels with little or no fines	Poorly graded sands with little or no fines	Silty Sand

3.5 Tensile strength test

The collection of exhumed samples and tensile strength tests should be conducted within 48 hours after the installation.

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The tensile strength test can be conducted as per the ASTM D6637-1 2016 Method A. Tensile strength test results for the both control specimen and exhumed specimens gives the reduction factor i.e., the installation damage factor.

4. Conclusion

It is concluded from the literature review that an installation damage test is necessary for the geotextiles since it will evaluate the damage factor of the geo-textiles.

In India, IRC SP 102 provided the importance of the installation damage tests for the geo-textiles. The installation damage test is one of the most sophisticated tests to be conducted on the geo-textiles and it is observed that test facilities are limited due to the complications in performing the test. Nowadays many geotextile industries, contractors, and agencies are willing to perform the installation damage test to evaluate the damage factor criteria of the geo-textiles because this will estimate the long-term performance of the geo-textiles. At the BTRA the test facilities will be developed to conduct the installation damage test as per the standard specifications. It is seen from many researchers that the installation damage is invertible however, it can be minimized to a greater extent by following standard operating procedure and careful supervision.

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Method Development for Analyzing Hexavalent Chromium in Water Soluble Dyes and its Validation

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Abstract

Hexavalent chromium present in water-soluble dyes is eluted through an activated carbon solid-phase extraction cartridge at a pH of 12 – 13. Dye chromophores, which interfere with the colourimetric detection of hexavalent chromium are removed by absorbing on the activated carbon bed. Un retained Cr(VI), eluted through activated carbon solid phase extraction system will give a purple-violet colour with Diphenylcarbazide reagent. Cr(VI) oxidizes 1,5 diphenylcarbazide to 1,5 diphenylcarbazone to give a purple /violet complex, which can be quantified spectrophotometrically at 540 nm wave length. The method described is suitable to quantify chromium (VI) content in water-soluble dyes down to 3 mg/kg. This in house developed test method has been validated as per international validation protocol for spike recovery at three different levels, repeatability (intra assay and intermediate precision), LOD, and LOQ for dyes like Reactive yellow HE 6G, Reactive red 218, Turquoise blue HGN, Reactive navy blue RX, Reactive black 5A.

KeyWords:

Activated carbon, Diphenylcarbazide, Hexavalent chromium, Inductively Coupled Plasma-Optical Emission Spectrometry, Spectrophotometer, Solid phase extraction.

1.0 Introduction

In its natural state, chromium mainly occurs in its trivalent [Cr(III)] and hexavalent[Cr(VI)] forms. Trivalent chromium is an essential micronutrient for humans and is nontoxic in normal doses in the food supply; at higher doses, however, trivalent chromium can exhibit cytotoxicity. Due to its high solubility and oxidizing property, hexavalent chromium is extremely toxic and has been confirmed to have a carcinogenic effect [1]. It is a well-established fact that once Cr(VI) reaches the bloodstream, it damages the kidney, liver, and blood cells of the human through oxidations reaction, and the patient leads to hemolysis, renal, and liver failure [2]. Cr(VI) leads to allergic reactions in human beings and even at low concentrations it can causes diseases such as dermatitis, ulceration, encephalopathy, anemia, hepatitis, and nephritic syndrome [3-5].Cr(VI) also possesses carcinogenicity and can damage the DNA of humans and animals [6].

Cr(VI) has a wide range of applications in industrial fields. A large number of chromium-based synthetic dyes are being increasingly used in the textile, paper, pharmaceutical, cosmetic, and food industries. It is also used in

electroplating, cement, steel, paint, dye, aluminum, leather tanning, metal finishing, and chromate manufacturing industries [7-8]. Thus, the effluents from these industries, when mixes with the soil or surface water create a health hazard to mankind. It is therefore needed to develop a method for environmental cleanup and remediation so that Cr(VI) is safely removed from the water. In the past few years, many techniques such as chemical precipitation, coagulation-flocculation, flotation, electrochemical, ultrafiltration, etc. have been used by various workers [9-12].

The total chromium content at trace levels is determined traditionally using graphite furnace atomic absorption spectroscopy (GFAAS) or by ICPOES.

The colourimetric method is widely used for the estimation of Cr(VI) in textiles, leather, drinking water, electronic components, etc.[13-15]. However, this method cannot be applied for the estimation of trace level of Cr(VI) in water soluble dyes, colored effluents, etc. due to the color interference while measuring the optical density of Cr(VI).

Simultaneous determination of trivalent and hexavalent chromium using accelerated solvent extraction and ion chromatography, LC-ICP-MS, etc. are some of the

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hyphenated techniques being used to analyze Cr(VI) [16-17]. Research work has also been carried out to separate Cr(VI) from water soluble dyes by HPLC pre-column as well as post column derivatization method [18-19]. However, all these instruments are very costly and many of the small-scale testing laboratories may find it difficult to invest a huge amount for costly instruments. Hence, it is the need of the hour to develop a simple and economically viable test method to quantify trace levels of Cr(VI) in water-soluble dyes.

In this study, we have explored various techniques to segregate Cr(VI) from dye chromophore, out of which, an activated charcoal bed was found to be the best absorption media for separation. The method involves a simultaneous process of absorption of dyes in an alkaline phase-activated charcoal and elution of hexavalent chromium.

2.0 Material and Method

2.1 Materials

- Pipette of capacities 1ml, 10ml
- Glass beaker of capacities 10 ml
- Volumetric flask of capacities 25ml, 100ml, 1000 ml
- Spectrophotometer wave length 540 nm
- pH meter in the range of 1-14
- Vacuum manifold connected to vacuum pump.
- Solid Phase Extraction (SPE) Column (3.0 g of activated charcoal in a 5.0 ml capacity syringe column)

2.2 Reagents

- Diphenylcarbazide solution (1.0g l, 5-diphenylcarbazide (DPC) in 100 ml acetone and acidified with one drop of glacial acetic acid.
- O-phosphoric acid solution (88%)
- Nitric acid (69%)
- Hydrofluoric acid (48%)
- Hydrochloric acid (37%)
- NaOH (3.0 % solution)
- Na₂CO₃ (0.5 % solution)
- Eluent mixture (50 ml 3% NaOH and 50 ml 0.5 % Na₂CO₃), pH 12-13
- Distilled water, Grade 3 quality as specified in ISO 3696
- Activated charcoal, MERCK (7440-44-0) particle size 100 mesh
- Potassium dichromate (K₂Cr₂O₇), dried for 16 hours at 102°C
- Cr(VI) stock solution: Dissolve 2.829 g K₂Cr₂O₇ (pre-dried for 16.0 hrs. at 102°C ±2°C) in distilled water and makeup to 1000 ml. 1 ml of this solution contains 1mg of Cr(VI) (1000 ppm).
- Cr(VI) standard solution: From stock solution pipette out 1 ml into a 100 ml volumetric flask and makeup to the

mark with distilled water. 1 ml of this solution contains 0.01 µg of Cr(VI) (10 ppm)

2.3 Instruments

- Analytical balance of accuracy 1 mg (Shimadzu AUX 220)
- pH meter, with a glass electrode (ELICO LI127)
- Microwave Digestion System (Anton Paar Multiwave GO)
- UV-VIS Spectrophotometer (Shimadzu UV 1800)
- Inductive Coupled Plasma Optical Emission Spectrophotometer (ICP-OES) (Perkin Elmer Optima 8000)

2.4 Methods

2.4.1 Estimation of total chromium content in dyes

In principle, the trace metal content in any matrix can be estimated after being subjected to either conventional acid digestion or high temperature and high-pressure microwave-assisted digestion, depending on the nature of the matrix. The digested sample can be analyzed by AAS/ICP-OES to quantify the metals. To estimate the total chromium (Cr(III) & Cr(VI)) in dyes, a known weight of the sample was digested using 10ml nitric acid in a microwave digestion system. The digested sample was diluted up to a certain volume and the total chromium content was determined using ICP-OES.

2.4.2 Estimation of Cr(VI) in dyes

Since Cr can undergo a redox reaction, it can easily oxidize to Cr(VI) and can reduce to Cr(III), care should be taken while the digestion/extraction process. All oxidizing agents and reducing agents should be avoided during the extraction/digestion process. By keeping this in mind, we have made various trials to segregate the Cr(VI) from dyes. For this purpose, first, we have studied the effect of different acids and alkalis on Cr(VI) during the digestion/extraction process.

2.4.2.1 Conventional acid digestion method

A known concentration of Cr (VI) solution was treated with different acids and digested using a conventional hot plate digestion method. After digestion, the solutions were derivatized with diphenylcarbazide and o-phosphoric acid, and color was developed. The optical density of the solution was measured at 540nm using UV-VIS Spectrophotometer and compared against standard.

2.4.2.2 Microwave assisted acid digestion method

Microwave digestion techniques are used to digest the toughest sample, as it works at high temperature (200-250°C) and high pressure (20-40 bar). To study the impact of various acids at high temperature and pressure on Cr(VI), a known concentration of Cr (VI) solution was digested with various acids in a microwave digestion system. After digestion, the

solutions were derivatized with diphenylcarbazide and o-phosphoric acid, and colour was developed. The optical density of the solution was measured at 540nm using UV-VIS Spectrophotometer and compared against standard.

2.4.2.3 Alkaline digestion method

Literature survey reveals that Cr(VI) is stable at alkaline pH and standard method like EPA recommends alkaline extraction for estimation of Cr(VI) from the soil. However, the efficacy of alkaline extraction for the estimation of Cr(VI) from dyes has not been studied. To study the alkaline extraction efficacy of Cr(VI) from dyes, a known concentration of hexavalent chromium was taken in a 100 ml conical flask. 10 ml of alkaline solution (2% of NaOH and 3% Na₂CO₃) was added to it. The solution was digested on a hot plate; then the solution was maintained at this temperature for 30mins. The flask was cooled to room temperature and the solution was derivatized using diphenylcarbazide and o-phosphoric acid and color were developed. The optical density of the solution was measured at 540 nm using UV-VIS Spectrophotometer and compared against standard.

2.4.2.4 Activated charcoal SPE method

- Preparation of SPE column

3.0 gram of activated charcoal powder (2.2.10) was weighed and filled in a 5.0 ml capacity syringe column. Before filling activated carbon powder, 4-5 layers of filter paper/nylon membrane were added to support the fine carbon particles in the column. An activated charcoal column was connected to a vacuum manifold (2.1.6). The column was washed 2-3 times with an eluent mixture (2.2.8) under vacuum.

- Analysis of Cr(VI) standard solution

1.0 ml of Cr(VI) solution (2.2.13) was loaded into a pre washed activated charcoal solid phase extraction cartridge and allowed to absorb in the column. Cr(VI) was eluted from the column with 20.0 ml of eluent mixture (2.2.8). The extract was collected in a 25.0 ml volumetric flask and colour was developed with 1.0 ml phosphoric acid (2.2.2) and 1.5 ml DPC solution (2.2.1). The final solution was made up to the mark with distilled water. The absorbance of the solution was measured at 540 nm wave length by using a spectrophotometer. The absorbance of this solution will be equivalent to 0.4 ppm concentration of Cr(VI).

- Analysis of dyes

0.5 g of dye was weighed in a clean glass beaker to the nearest 0.001g. The dye was dissolved in a 10 ml eluent mixture (2.2.8) and a stock solution was prepared.

The above entire stock solution was loaded directly into a pre washed SPE column (2.1.7) drop by drop and the solution was allowed to absorb throughout the column. The beaker was washed with a 5ml eluent mixture (2.2.8) and the solution was poured drop by drop on top of the column and Cr(VI) was eluted under vacuum. The extraction was repeated another 3 times with a 5.0 ml aliquot of eluent mixture. The entire extract (approximately 20ml) was collected in a 25.0 ml volumetric flask. (Dye molecules will absorb in activated carbon under alkaline pH and unretained

Cr (VI) will elute). The color was developed by adding 1.0 ml o-Phosphoric acid (2.2.2) followed by 1.0 ml 1,5 DPC solution (2.2.1) and the solution was made up to 25.0 ml with distilled water. This solution was kept for 5 minutes at ambient temperature and the absorbance was measured at 540 nm wave length by using a Spectrophotometer against a blank solution.

Note: The above weight of dye and volume of the extract is for the determination of Cr(VI) down up to 3.0 mg/kg. If the Cr(VI) content is high, either a less weight or a small aliquot from the stock solution may be taken so that absorbance value is within the calibration range of Cr(VI).

- Blank solution

The eluent mixture (2.2.8) was filled three quarters in a 25ml volumetric flask and 1.0 ml phosphoric acid (2.2.2) followed by 1.0 ml 1,5 DPC solution (2.2.1) was added and makeup to 25 ml.

2.5 Calculation

Calculate the Cr(VI) content in dyes as per the given formula

$$w_{Cr(VI)} = \frac{C \times V}{m}$$

Where,

wCr(VI) is the mass fraction, expressed in milligram per kilogram (ppm) of soluble Cr(VI) in dyes.

C is the concentration of Cr(VI) obtained from the calibration graph of standard (mg/kg)

m is the mass of the dye sample taken, expressed in grams (g)

V is the dilution factor (ml)

3.0 Results and Discussion

Total chromium content in various commercial dyes analyzed by ICP OES is tabulated in Table 1. It has been observed that few dyes like Direct blue GL, Reactive Black, Basic Brown, etc. do not contain any chromium, whereas, dyes like XP-20 N, disperse black, etc. contain a high amount of chromium. The total chromium content in XP-20N is 1.55% and disperse black is 0.15%. Since these are total chromium (Cr (III) and Cr(VI)), a good separation technique is needed to segregate and estimate hexavalent chromium.

Table 1 Total chromium content in Dyes

Dyes	Chromium (mg/kg)
Direct blue GIL	ND
Reactive black	ND
Remazole black	ND
Basic brown	ND
Solvent brown	ND
Solo blue B	ND
Solo blue T	ND
Reactive orange	4.0
XP-20 N	15570
Disperse black	1556

Various acids are used for the digestion of Dyes to estimate total chromium by conventional as well as microwave digestion methods. But the effect of these acids on Cr(VI) under various temperatures and pressure has not been studied. Hence, we have conducted a study on the effect of acids on Cr(VI). Table 2 shows the effect of various acids on hexavalent chromium under conventional digestion.

From Table 2, it was observed that digestion of Cr (VI) by a conventional method is not suitable, as all the acids interfere either with Cr (VI) or with DPC. When hexavalent chromium was digested by a conventional method, without acid (only with water) purple/violet color of Cr (VI) developed and the color was stable for a long duration. This indicates that most of the acids have an adverse impact on Cr(VI).

Table 2 - Effect of various acids on Cr(VI) in a Conventional digestion

Conc. Of Cr (VI)	0.4mg/kg	0.4mg/kg	0.4mg/kg	0.4mg/kg
Acids	1ml HNO ₃	1ml HCl	1ml HF	1ml Water
Method	Digestion in hot plate followed by colour development			
Visual Observation	The purple colour of Cr(VI) is not Stable	The purple colour of Cr(VI) is not Stable	The purple colour of Cr(VI) is not Stable	Purple colour of Cr(VI) Stable

Table 3 shows the effect of various acids on Cr(VI) at high temperature and high-pressure microwave digestion system.

When a Standard solution of Cr(VI) was digested in a microwave digestion system without any acid (only with water) purple color of Cr (VI) developed and the color was stable for a long duration. At the same time, when the same standard solutions were treated with various acids and derivatized with DPC, the color was either not developed or the developed colour was not stable. The same effect was observed in a hot plate conventional acid digestion also. This indicates that no acids can be used for the analysis of Cr(VI) as it adversely affects the optical density measurement.

Table 3 - Effect of acids on Cr(VI) by Microwave digestion system

Conc. Of Cr(VI)	0.4mg/kg	0.4mg/kg	0.4mg/kg	0.4mg/kg
Acids	9ml HNO ₃	9ml HCl	9ml HF	9ml Water
Method	By Microwave Digestion followed by colour development			
Visual Observation	No Purple colour	No Purple Colour	No Purple colour	Purple colour Stable

Table 4 shows the effect of alkaline extraction on Cr(VI) based dyes. The results indicate that hexavalent chromium

can be extracted with the alkaline digestion method with a recovery of 90%. However, this method is not suitable for analysis of Cr(VI) in water soluble dyes as in alkaline extraction the color of the dye (chromophore) will also co elutes along with Cr(VI).

Since hexavalent chromium is having good stability at alkaline pH, few extraction trials were conducted in an alkaline medium. It was found that activated charcoal, at an alkaline pH can absorb all types of water soluble dyes, at the same time it doesn't have any affinity to Cr(VI). Based on this finding, a study was carried out by spiking a concentration of 5.0mg/kg standard Cr(VI) in various water soluble dyes which were free from chromium. The dye solution spiked with Cr(VI) was loaded into an activated carbon bed at pH 12 to 13. The substance which interferes with the detection (dye chromophores) is removed by absorbing in the activated carbon bed and unretained Cr(VI) was eluted. The resultant eluent was derivatised and the developed color was measured in a UV VIS spectrophotometer at 540nm wavelength. The spike recovery thus obtained is tabulated in Table 5. The Recovery of Cr(VI) was found to be more than 90 % in all dyes. This indicates that estimation of Cr(VI) by activated carbon in alkaline pH can be used to segregate hexavalent chromium from interfering dye chromophores. Based on the spike recovery study, further, the method has been validated as per the International validation protocol.

Table 4 Effect of Alkalis on Cr(VI) based dyes

Samples	Standard Cr(VI)	Reactive Black dyes spiked with Cr(VI)	Basic Brown spiked with Cr(VI)
Cr(VI) Concentration	0.4mg/kg	0.4mg/kg	0.4mg/kg
Method	Alkaline digestion followed by colour development		
Visual observation	Purple colour	Co-elution of dye colour (Black)	Co elution of dye colour (Brown)
Measured Concentration	0.360 mg/kg	Measurement not possible	Measurement not possible
Recovery (%)	90	-	-

Since hexavalent chromium is having good stability at alkaline pH, few extraction trials were conducted in an alkaline medium. It was found that activated charcoal, at an alkaline pH can absorb all types of water soluble dyes, at the same time it doesn't have any affinity to Cr(VI). Based on this finding, a study was carried out by spiking a concentration of 5.0mg/kg standard Cr(VI) in various water soluble dyes which were free from chromium. The dye solution spiked with Cr(VI) was loaded into an activated carbon bed at pH 12 to 13. The substance which interferes with the detection (dye chromophores) is removed by absorbing in the activated carbon bed and unretained Cr(VI) was eluted. The resultant eluent was derivatised and the developed color was measured in a UV VIS spectrophotometer at 540nm

wavelength. The spike recovery thus obtained is tabulated in Table 5. The Recovery of Cr(VI) was found to be more than 90 % in all dyes. This indicates that estimation of Cr(VI) by activated carbon in alkaline pH can be used to segregate hexavalent chromium from interfering dye chromophores. Based on the spike recovery study, further, the method has been validated as per the International validation protocol.

Water soluble dyes	Spiked Conc. (mg/kg)	Replicates of Recovered Concentration(mg/kg)						Recovery (%)
		1	2	3	4	5	Mean	
Yellow HE 6G	5.0	4.92	4.64	4.88	4.76	4.88	4.81	96.2
Reactive Red 218	5.0	4.80	4.88	4.73	4.64	4.80	4.77	95.4
Reactive black 5A	5.0	4.60	4.72	4.50	4.57	4.68	4.61	92.2
Navy Blue RX	5.0	4.60	4.84	4.58	4.53	4.76	4.66	93.2

4.0 Method Validation

The developed test method has been validated as per Eurachem and ICH guidelines for the determination of Accuracy, Precision, Reproducibility, Linearity, LOD & LOQ.

4.1 Accuracy

The accuracy of an analytical procedure expresses the closeness of agreement between the value that is accepted either as a conventional true value or an accepted reference value and the value found. This is sometimes termed trueness. For the quantitative approaches, accuracy can be determined by spiking three levels and at least 10 replicates should be obtained. The percentage recovery or the difference between the mean and the accepted true value together with the confidence levels are recommended. For accuracy, the mean recovery within 90-110% of the theoretical value may be taken as acceptance criteria.

To determine the spike recovery, a known amount of three different concentrations i.e. 5mg/kg, 10mg/kg, 20mg/kg of Cr(VI) standard solution was spiked in five different dyes. The spiked samples were analyzed as per the test method and the spike recovery was calculated. Ten replicates were taken to calculate the statistical parameters like SD and RSD. The results are tabulated in Table 6A, 6B, & 6C.

From Table 6A it can be seen that the Spike recovery of all dyes is above 90%. Similar spike recovery is shown in 10 mg/kg and 20 mg/kg levels also. The percentage relative standard deviation (RSD) of recovery is below 5.0%.

Table 6A Spike recovery of Cr(VI) at 5 mg/kg

Table 6A Spike recovery of Cr(VI) at 5 mg/kg

Dyes #	Yellow HE 6G	Reactive Red 218	Turquoise Blue HGN	Navy blue RX	Reactive Black 5A
1	98.4	96	98.4	92	92
2	92.8	100	96.8	96.8	94.4
3	97.7	94.6	96.9	91.6	90.2
4	95.3	96	97.6	90.6	91.4
5	97.6	92.9	88.9	95.2	93.7
6	94.6	97.6	93	96.1	91.5
7	96.1	91.6	93.1	96.1	90
8	91.4	93.7	96.8	99.2	96
9	95.3	92.9	90.6	96	93.7
10	89	100	95.3	92.9	98.4
Mean (%)	94.82	95.53	94.74	94.65	93.13
SD (ppm)	2.99	2.95	3.20	2.74	2.66
RSD (%)	3.16	3.09	3.37	2.89	2.85

Table 6B Spike recovery of Cr(VI) at 10 mg/kg

Dyes #	Yellow HE 6G	Reactive Red 218	Turquoise Blue HGN	Navy Blue RX	Reactive Black 5A
1	92.3	93.1	90.7	93.5	94.3
2	94.4	93.8	86.1	98.3	95
3	94.7	97.1	97.9	98.3	95.1
4	97.7	102	95	93.6	97.7
5	95.4	94.1	90	90	90.5
6	86.7	92.6	93.7	93.3	99.2
7	96.1	94.5	91.8	91.8	93.3
8	91.2	94.4	95.2	95.6	94.4
9	96.8	96	94.1	90.9	91.7
10	98.4	92.4	93.6	96.8	98.8
Mean(%)	94.37	95	92.81	94.21	95
SD (ppm)	3.50	2.86	3.30	2.95	2.87
RSD(%)	3.71	3.01	3.55	3.13	3.02

Table 6C Spike recovery of Cr(VI) at 20 mg/kg

Dyes #	Yellow HE 6G	Reactive Red 218	Turquoise Blue HGN	Navy Blue RX	Reactive Black 5A
1	97.2	92.1	90.5	91.3	90.5
2	93.9	94.3	91	93.5	98.3
3	99.2	90.1	95.6	93.6	90.9
4	89.8	94.9	87.1	86.7	87.9
5	95.5	91.5	91.9	93.9	91.9
6	95.5	91.2	97.5	98.3	94.3
7	96.4	90.9	90.5	92.9	94.1
8	96.1	94.2	92.2	91.4	90.6
9	94.9	94	97.6	99.6	98.8
10	97.4	94.6	90.5	98.6	95.9
Mean (%)	95.59	92.78	92.44	93.98	93.32
SD (ppm)	2.51	1.79	3.41	3.94	3.58
RSD(%)	2.63	1.93	3.69	4.20	3.84

4.2 Precision

The precision of an analytical procedure expresses the closeness of agreement (degree of scatter) between a series of measurements obtained from multiple sampling of the same homogenous sample under the prescribed conditions. Precision (measurement precision) is a measure of how close results are to one another. It is usually expressed by statistical parameters which describe the spread of results, typically the standard deviation (or relative standard deviation) calculated from results obtained by carrying out replicate measurements on a suitable material under specified conditions. Precision may be considered at three levels: repeatability, intermediate precision, and reproducibility.

4.2.1 Repeatability (Intra-assay precision)

Repeatability, expected to give the smallest variation in results, is a measure of the variability in results when a measurement is performed by a single analyst using the same equipment over a short timescale. Repeatability expresses the precision under the same operating conditions over a short interval of time. To determine the repeatability, a known amount of Cr(VI) was added to five different dyes. This test was carried out for six different days and the mean, SD, RSD was calculated. The results are tabulated in Table 7

Table 7 Repeatability of Cr(VI) in dyes at 10 mg/kg

Dyes	Yellow HE 6G	Reactive Red 218	Turquoise Blue HGN	Navy Blue RX	Reactive Black 5A
Days					
Day-1	9.23	9.31	9.07	9.35	9.43
Day-2	9.44	9.38	8.61	9.83	9.5
Day-3	9.47	9.71	9.79	9.83	9.51
Day-4	9.77	10.2	9.5	9.36	9.77
Day-5	9.92	9.68	9.02	8.91	8.25
Day-6	9.12	9.44	9.52	9.56	9.44
Day-7	9.79	9.79	9.38	9.58	9.54
Mean (mg/kg)	9.53	9.64	9.27	9.49	9.35
SD(ppm)	0.30	0.31	0.39	0.32	0.50
RSD(%)	3.17	3.16	4.26	3.38	5.32

From Table 7, the data shows that the RSD % of repeatability of all dyes is below 5% which is within the acceptable limits.

4.2.2 Intermediate precision

Intermediate precision expresses within laboratories variation: different days, different analysts, different equipment, etc. Intermediate precision gives an estimate of the variation in results when measurements are made in a single laboratory but under specified conditions that are more variable than repeatability conditions. The aim is to obtain a precision estimate that reflects all sources of variation that will occur in a single laboratory under routine conditions (different analysts, extended timescale, etc.)

To determine the intermediate precision, a known amount of

Cr(VI) was spiked in five different dyes. Cr(VI) in these dyes was measured on different days by seven different analysts and the mean, SD, RSD was calculated. The results are tabulated in Table 8.

Table 8 Intermediate precision of Cr(VI) at 10mg/kg

Operator	Dyes	Yellow HG 6E	Reactive Red 218	Turquoise Blue HGN	Navy Blue RX	Reactive Black 5A
	Days					
A	Day-1	9.47	9.71	9.79	9.83	9.51
B	Day-2	9.54	9.41	9	9	9.05
C	Day-3	8.67	9.26	9.37	9.33	9.92
D	Day-4	9.61	9.45	9.18	9.18	9.33
E	Day-5	9.68	9.6	9.41	9.09	9.17
F	Day-6	9.84	9.24	9.36	9.68	9.88
G	Day-7	9.51	9.27	9.35	9.47	9.23
	Mean	9.47	9.42	9.35	9.37	9.44
	SD(ppm)	0.38	0.18	0.24	0.31	0.34
	RSD(%)	3.97	1.93	2.58	3.29	3.64

Table 8 shows that the intermediate precision of all dyes tested at 10 mg/kg level is below 5 % RSD which is within the acceptable limit.

4.2.3 Reproducibility (Inter-Laboratory Comparison)

Reproducibility represents the precision obtained between different laboratories. The objective is to verify that the method will provide the same results in different laboratories. The reproducibility of an analytical method is determined by analyzing aliquots from homogeneous lots in different laboratories with different analysts and by using operational and environmental conditions that may differ from, but are still within, specified parameters of the method (interlaboratory tests). Reproducibility, expected to give the smallest variation in results, is a measure of the variability in results between laboratories. To estimate the reproducibility of the test method, two dyes samples that contain Cr(VI) were sent to seven accredited testing laboratories along with the in-house developed test method. The round-robin test results were collected and the robust Z score of each laboratory was calculated. The ILC results are tabulated in Table 9.

Table 9 data shows that the robust Z score value of all the eight laboratories is within the acceptable limit of below ± 2.0 .

Table 9 Reproducibility of Cr(VI) in water soluble dye

Lab code	Dye		Dye	
	Reactive red	Z	Reactive blue	Z
	Cr(VI) (mg/kg)	Score	Cr(VI) (mg/kg)	score
01	9.69	-0.77	9.38	-1.32
02	9.5	-1.48	9.8	0.10
03	9.79	-0.39	10.2	1.46
04	10.31	1.56	9.92	0.51
05	10.07	0.66	10	0.78

Lab code	Dye		Dye	
	Reactive red	Reactive blue	Reactive red	Reactive blue
	Cr(VI) (mg/kg)	Z Score	Cr(VI) (mg/kg)	Z score
06	10.04	0.54	9.6	-0.58
07	9.64	-0.96	9.49	-0.95
07	9.74	-0.58	9.74	-0.10
07	10.1	0.77	9.4	-1.26
08	10	0.39	9.9	0.44
Median (Q2)	9.89		9.77	
Lower Quartile (Q1)	9.70		9.52	
Upper Quartile (Q3)	10.06		9.92	
IQR	0.36		0.40	
IQR X 0.7413	0.27		0.29	

4.3 Linearity

The linearity of an analytical procedure is its ability (within a given range) to obtain test results that are directly proportional to the concentration of analyte in the sample. Linearity may be directly demonstrated on the analyte, or spiked samples using at least five concentrations over the whole working range. Evaluation of the analyte signal as a function of the concentration, appropriate statistical calculations are recommended, such as linear regression.

To plot a calibration graph of Cr(VI) standard solution, the first 1000 mg/kg of stock solution was prepared by exactly weighing 2.8292 grams of dried $K_2Cr_2O_7$ in 1000 ml distilled water. From this 1000 ppm solution, an intermediate solution of 10 mg/kg was prepared. From 10 mg/kg of standard solution a series of calibration solutions were prepared by pipetting out 0.5ml, 1.0 ml, 2.0 ml, 3.0 ml, 4.0 ml, 5.0 ml, and 6.0 ml into seven 25 ml volumetric flasks. These flasks were three quarters filled with distilled water and 0.5 ml H_3PO_4 (2.2.2) and 0.5 ml DPC (2.2.1) solutions were added and makeup to the mark. The concentration of Cr(VI) in these solutions will be 0.2,0.4,0.8,1.2,1.6, 2.0 and 2.4 mg/kg respectively. The optical density of these colored solutions was measured at 540 nm wave length against a blank reading. A seven-point calibration graph was plotted, absorption verses concentration. Figure 1 represents a linear regression graph of Cr(VI) when plotted in the range of 0.2 to 2.4 mg/kg at 540 nm wave length. The regression coefficient (r^2) of the calibration graph was 0.9990 which indicates Cr(VI) solution gives good linearity over a range of 0.2-2.4 mg/kg.

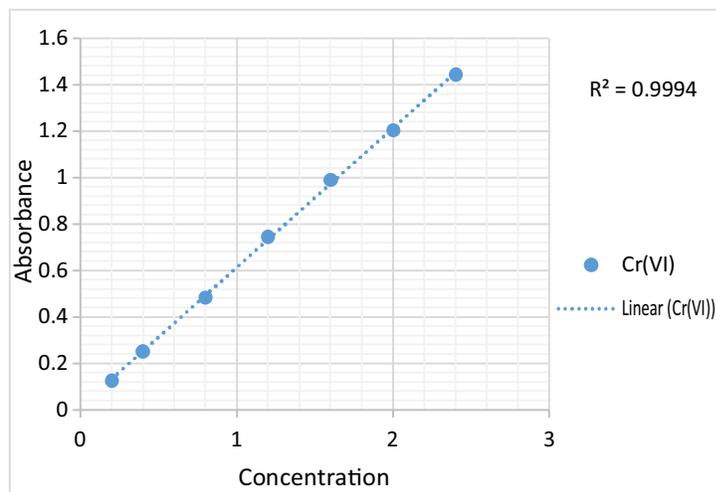


Figure 1 Calibration graph of Cr(VI)

4.4 Range

The specified range is normally derived from linearity studies and depends on the intended application of the procedure. The range of an analytical method is the interval between the upper and lower levels that have been demonstrated to be determined with precision, accuracy, and linearity using the method as written. The range is normally expressed in the same units as the test results. From figure 1 it can be seen that Cr(VI) gives good linearity in the working range of 0.2-2.4 mg/kg.

4.5 Limit of Detection (LOD)

LOD can be defined as the lowest amount of analyte in a sample which can be detected but not necessarily quantitated as an exact value. This is also known as the instrument detection limit. The instrument detection limit can be calculated based on a standard deviation of the response based on the standard deviation of the blank or standard deviation of the response based on the slope of the calibration curve. A specific calibration graph (Figure.1) is studied using samples containing analyte in the range of limit of detection. The residual standard deviation of the y-intercepts of regression lines may be used as the standard deviation.

4.6 Limit of Quantification (LOQ)

LOQ can be defined as the lowest amount of analyte in a sample which can be quantitatively determined with suitable precision and accuracy. This is also known as the method detection limit.

Table 10 shows IDL and MDL of Cr(VI) by using a Spectrophotometer calculated based on a calibration graph. This indicates that a spectrophotometer can detect down up to 0.07mg/kg Cr(VI). Hence, by considering the weight of dye and final volume of extract, Cr(VI) in dyes can be quantified down up to 3.0 mg/kg.

Table 10 LOD of Cr(VI)

Calibration standards	Concentration (mg/l) X axis	Absorbance Y axis
Std-1	0.2	0.127
Std-2	0.4	0.254
Std-3	0.8	0.254
Std-4	1.2	0.746
Std-5	1.6	0.993
Std-6	2.0	1.204
Std-7	2.4	1.445
Regression coefficient (R ²)	0.9994	
The slope of the curve (M)	0.60008	
Std error (STE)	0.012756	
LOD=(STE/M) *3.3	0.07015	

4.7 Measurement Uncertainty

Uncertainty is a parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand. When estimating the uncertainty of measurement, all uncertainty components which are important in the given situation shall be taken into account using appropriate methods of analysis. Measurement uncertainty, in general, has many components. Some of these components may be evaluated from the statistical distribution of the results of a series of measurements and can be characterized by experimental standard deviation. Other components, which also can be characterized by standard deviations, are evaluated from assumed probability distributions based on experience or other information. The calculated measurement uncertainty

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Table 11 Measurement Uncertainty of Cr(VI) in different dyes

Dyes	Reactive Red 218	Yellow HE 6G	Turquoise Blue HGN	Navy Blue RX	Reactive Black 5A
Measurement uncertainty (MU) mg/kg	At 9.49 ppm ±0.16 ppm	At 9.63ppm ±0.13ppm	At 9.35ppm ± 0.26ppm	At 9.29ppm ±0.29ppm	At 9.40ppm ±0.30ppm

(MU) of Cr(VI) in five dyes at 10 ppm level is given in Table 11. (Since the measurement uncertainty calculation involves a lot of statistical parameters, each step has not been elaborated here. The only final calculated value mentioned). The reactive red dye which contain 10 ppm Cr(VI), measured concentration is 9.49 ppm and can be reported with an accuracy ranging from 9.33 to 9.65 ppm based on the measurement uncertainty. Similarly Reactive black at 9.4 ppm level, accuracy will be in the range of 9.1 to 9.7 ppm.

5.0 Conclusions

Chemical speciation of Cr(VI) in water soluble dyes is not possible either by microwave digestion or by conventional digestion method as Cr (VI) reacts with all acids and loses at high temperature. The potential of activated carbon for the adsorption of dye molecule and recovery of Cr(VI) in an alkaline medium has been explored in this test method. This test method was found to be one of the best economical methods for the determination of Cr(VI) in water soluble dyes. By this method Cr(VI) in water soluble dyes were found to have good repeatability and reproducibility. The round robin test conducted with seven laboratory results agrees that the method has good reproducibility. By this method Cr(VI) in water soluble dyes can be quantified down up to 3.0mg/kg level.

Hence, this in-house developed and validated test method can be used to test Cr(VI) in all types of water soluble dyes.

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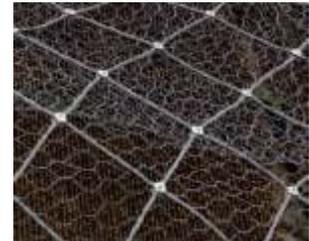
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