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AN EXPERIMENTAL STUDY TO DETERMINE THE IMPACT OF SEQUESTERING AGENT IN TEXTILE WET PROCESSING

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***Abstract**: Sequestering agents can be used at most stages of textile wet processing of fibres, yarns, and fabrics. The general aim is to chemically remove, render, inert, any metallic ions that can interrupt further processing. This also includes the alkaline earth metals, such as calcium, magnesium. The emphasis as to which ions are required to be sequestered depends upon the process taking place .Use of sequestering agent have the advantage of aiding in the dispersion of by-products in bleaching and prevention of dye aggregation. From our study we have seen that with the increase of pH in bleaching the performance of sequestering agent reduced significantly .It is also observed that when dosing of sequestering agent increase beyond a level the CIE whiteness value decreases. For water correction with the increase of dosing of sequestering agent, hardness gets down.*

***Keywords**: Sequestering agent, Chelation, Stabilize, Hardness, Binding capacity, Bleaching, pH, ppm.*

***Introduction:** Bleaching is the second steps of pretreatment of textile materials as well as wet processing technology as the first one is scouring. But today commercial scouring-Bleaching is done in single stage. Bleaching process can be defined as; the Destruction of natural coloring matter from the textile materials in order to achieve a clean white end product. For single stage cotton scouring-bleaching some basic chemicals as well as some auxiliaries are essential such as- Caustic Soda, Hydrogen Peroxide, Stabilizer, Detergent, Sequestering agent. Each of the chemicals have its own function for successful processing.*

Figure: Cotton ball

***Discussion:** Generally sequestering agent is used in textile wet processing for minimizing the detrimental effect of calcium, magnesium, iron ions what interrupt the optimum operation to take place. The amount of the presence of calcium, magnesium salts is termed as hardness. The metallic salts are present in* *water in two forms – carbonates & sulphates. The sulphates are responsible for permanent hardness whereas carbonates for temporary hardness.*

$$Ca(HCO_3)_2 \xrightarrow{\Delta} CaCO_3\downarrow + CO_2\uparrow + H_2O$$

$$Mg(HCO_3)_2 \xrightarrow{\Delta} MgCO_3 + CO_2\uparrow + H_2O$$

$$\rightarrow Mg(OH)_2\downarrow + CO_2\uparrow$$

$$2C_{17}H_{33}COOK + CaCO_3 \xrightarrow{\Delta} (C_{17}H_{33}COO)_2Ca\downarrow + K_2CO_3$$

In case of bleaching,if proper bleaching does not take place it will affect the subsequent processing of dyeing & finishing .In that case uneven shade, color spots, dye wastage will appear during dyeing process.There are

some types of sequestering agents according to the pH such as- acidic type, neutral to alkaline type. The acidic type sequestering agents are proton donor & cannot be used in dye bath.

The classification may be in the other way that is- sequestering agent for masking Iron & sequestering agent for calcium. Some sequestering agent are better for masking Iron ion where as some are used for masking calcium ions. The capacity of sequestering agent is generally denoted by the binding capacity of calcium and the binding capacity if Iron. There are totally different methods for calculating the binding capacity of calcium and the binding capacity of Iron ions of sequestering agent. The capacity is generally expressed as ppm (parts per million).1^oDh=17.848 ppm.

Main chemical classes of sequestrant [2]:

The five main classes of sequestrant used in the textile industry are:

- √ *Polyphosphonic acid.*
- √ *Amino polycarboxylic acid.*
- √ *Polyphosphates.*
- √ *Hydro carboxylic acid.*
- √ *Polymeric carboxylic acid.*

Characteristics of sequestering agents [2]:

- *Different sequestering power. (i.e, the strength of the sequester - metal complex)*
- *Different specific sequestering power for individual metal.*
- *Sequestering power not only depends on the specific sequestrant / metal pairing, but also on pH and temperature.*
- *Different sequestering capacities.*
- *Sequestering capacities which are metal ion specific.*
- *Difference in the spread of pH over which a particular sequestrant will combine with a particular metal ion.*
- *That not all sequestrants are stable to high temperatures.*

●That not all sequestrants are stable to oxidation or hydrolysis.

●That some sequestrants will de-metalize/pre-metalize dyes.

●That some sequestrants can affect the shade reproducibility of some dyestuffs (the effects can be very dye-sequestrant specific).

●That some sequestrant does not satisfy the environmental and toxicological requirements of every market

Structure of sequestering agent:

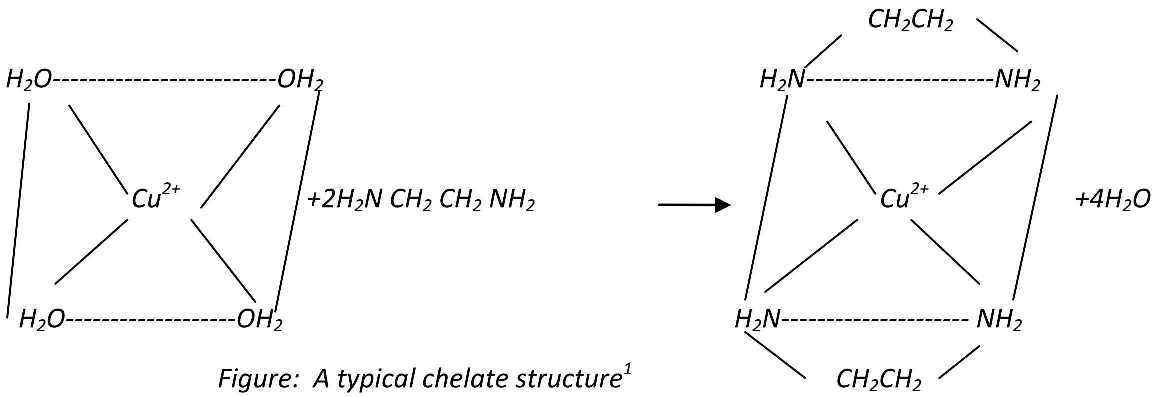

Figure: A typical chelate structure[1]

The positively charged metal ions particularly Fe^{3+} and Ca^{2+} are readily available for reaction with any negatively charged anion such as OH^- or CO_3^{-2} and insolubilize soap in the fiber which may disturb subsequent operation. This problem is much more acute when scouring is carried out in continuous process.

Problems created by calcium and magnesium ions if not sequestered properly [2]

►If calcium and magnesium are not sequestered, there is the strong possibility of their combining with natural "soaps" which have been generated during the alkaline scouring process, to form waxy substance. These have been referred to as "Lime soap deposits" they can deposit not only on the substrate itself but also on the surface of machinery.

►Calcium and magnesium ions reduce the solubility of anionic dyes causing them to aggregate or even precipitate on the fiber.

►Aggregated and precipitated dyes cannot migrate or diffuse they remain on the fiber surface as particular deposits.

A chemical compound having two (or more) chemical groups, which can surround a metal ion, resulting into a complex is called chelate. These chelates are more stable than the simple salts formed with acid (i.e acetic or formic acid) such which

are usually highly soluble and useful way of demineralizing cotton before dyeing .

However, they are not always resistant to precipitating condition and also often easily stripped of the metal ion by a dyestuff molecule, obviously, this is the opposite of the effect required, one of the main class of sequestrant is polyphosphate.

Another most commonly used sequestrant of textile industry is EDTA, which is considered as most stable complex, because the metal atom is enclosed in a 5 or 6 member ring.

Sequestering agents or chelating agents are negatively charged & capable of forming strong ring structures with the metal ions present in hard water and pectin's of cotton.

Thus the function of the chelating agents in the soap & detergent formulations are for the prevention of [2]—

- *Film and scum formation*
- *Calcium and magnesium inhibition of foaming properties*
- *clogging of liquid dispersions*
- *Haze turbidity in liquid solutions and*
- *ran city and oxidation that cause discoloration of formulation.*

Sequestering power is influenced by pH of the bleaching bath. At a given pH, different amount of chelating agents are required to chelate a given amount of metal.

If proper amount of sequestering agent is not use in the recipe so many problems will arise—

Firstly, when the system is not stabilized correctly under the influence of metallic catalyst such as Iron, The reaction is heavily pushed towards the free radical route. This causes a high degree of degradation of the peroxide ultimately pin hole occur.

Free radical reaction

H_2O_2 -------------- > $2H_2O + O_2$

The interim reactions result in the production of the free radicals which are not selective and will react and damage the cellulosic molecules. This is happening because it is impossible to control the decay of Hydrogen Peroxide and it proceeds by the free radical route. This causes the cellulose molecules to be split into two many times. As a result of this the fibre in the area or iron contamination becomes very weak. As the fabric is stressed during processing especially the long dyeing phase the tensions cause the fabric to tear in the areas of high damage, resulting in holes. These are usually small hence the pin hole term but can be larger.

But if the system is balanced with proper amount of correct sequestering agent the undesirable catalytic damage will not occur & optimum bleaching result will be found.

Via Perhydroxyl ion:-

H_2O_2 -------------- > $HOO^- + H^+$

This is the reaction which occurs when the bleaching bath is well stabilized and results in fibre that is well bleached with a small amount of fibre damage.

Secondly, when the amount of sequestering agent will be higher than the optimum dosage, the peroxide will be over stabilized i.e optimum bleaching will not happen so to get required whiteness the recipe will have to be adjusted by an increased amount of hydrogen peroxide or need some amount of magnesium salts (magnesium sulphate , magnesium chloride).

Experiment:

1. ***<u>Effect of pH on activity of Sequestering Agent</u>*:**

√ Apparatus: Conical flask, Measuring cylinder, Digital pipette, Burette, PH meter, Digital balance , Beaker.

√ Reagents: Standard calcium acetate solution (44.1 g/l), Sodium hydroxide, Sodium carbonate, Distilled water.

√ Chemical: Sequestering agent
Procedure:

- *Weigh 10g of sequestering agent to be tested and dissolved in a 100ml flask which is then filled up to the mark.*
- *Take 10ml from this solution and dilute it with 90ml distilled water, adjust to pH 7-8 with 1N NaOH.*
- *Add 10ml of 2% Na_2CO_3 solution. Adjust the solution to pH 11, 12, 13 by adding 1N NaOH and the total volume will be increased to approximately 150ml.*
- *Titrate with Ca acetate solution until permanent turbidity is achieved. Titration is done by gradual addition and should not stir while adding. pH should be constant throughout the whole procedure and be corrected with 1N NaOH if necessary.*

Calculation:

- *Calcium binding capacity(ppm)= volume of titer in ml*25*

<u>Result evaluation-</u>

Name of sequestering agent	Type of sequestering agent	Ca2+ binding capacity (ppm) at pH 11	Ca2+ binding capacity (ppm) at pH 12	Ca2+ binding capacity (ppm) at pH 13
Sample 1	Organic phosphorous compound	210	180	145
Sample 2	Combination of Organic phosphorous compound & carboxylic acid compound	200	175	137

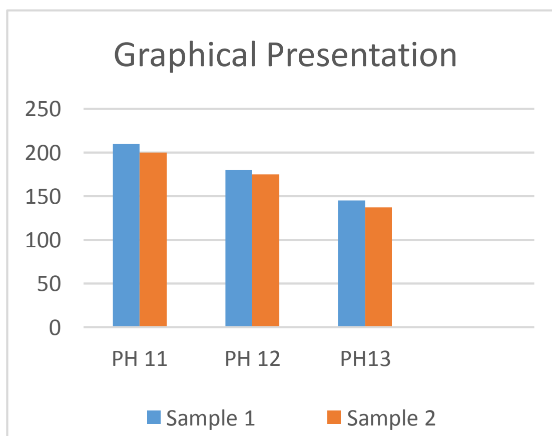

Figure: Effect of PH on Ca2+ binding capacity of sequestering

From the above experimental data it is very clear to understand that with the increase of pH the Ca^{2+} binding capacity of sequestering decreases. As chelation is a reversible reaction, the equilibrium is dependent on the process pH and the concentration of the metal ions and chelating agent, which react *together to form a chelate. The stability of the metal complex is expressed in terms of its stability constant. A high value of Ks indicates high sequestering effect.*

2. Effect of Sequestering Agent dosages on CIE whiteness index of bleached fabric:

√ Apparatus: Beaker, Digital pipette, Digital balance, measuring cylinder, scissor.

√ Chemicals: Detergent, Sequestering agent, stabilizer, NaOH, H_2O_2.

√ Machine: IR (infrared) lab dyeing machine

-Brand: Rapid

-Origin: Taiwan

√ Recipe:

Detergent: 0.6 g/l

Peroxide Stabilizer: 0.3 g/l

Sequestering agent: (x)

NaOH: 2 g/l

H_2O_2: 2 g/l

M:L 1:8

Bleaching @98℃X 40 MIN

<u>Result evaluation</u>:

Name of sequestering agent	*Type of sequestering agent*	*CIE whiteness index on 0.5 g/l Dosage in bleaching recipe*	*CIE whiteness index on 1 g/l Dosage in bleaching recipe*	*CIE whiteness index on 1.5 g/l Dosage in bleaching recipe*	*CIE whiteness index on 2 g/l Dosage in bleaching recipe*
Sample 1	*Organic phosphorous compound*	*57.31*	*58.38*	*54.42*	*51.73*
Sample 2	*Combination of Organic phosphorous compound & carboxylic acid compound*	*55.68*	*57.64*	*56.63*	*53.73*

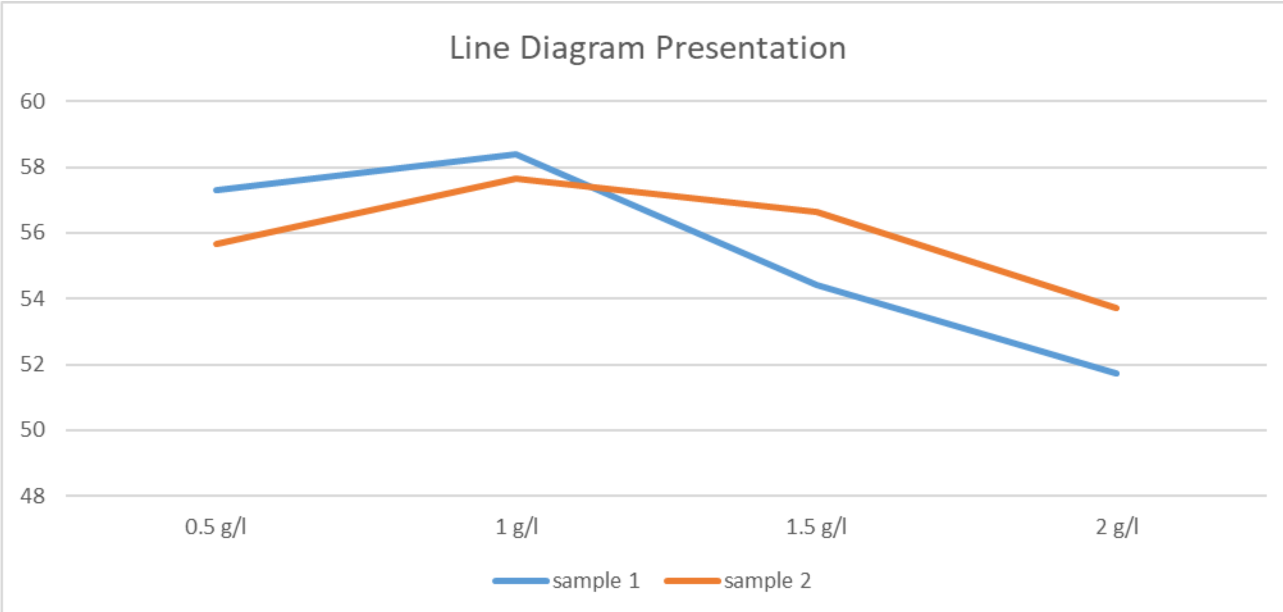

Figure: Effect of sequestering agent dosing on CIE whiteness index

From the above experimental data it is very clear to understand that with the increase of Sequestering agent dosage above optimum level the peroxide becomes over stabilized consequences lower CIE whiteness index value.so before put a recipe one should have to know the optimum dosages of sequestering agent for desired bleaching to take place.

3. Effect of Sequestering Agent on reduction of water Hardness:

√ Apparatus: Measuring cylinder, Beaker, Digital pipette, conical flask, Burette.
√ Reagents: Eriochrome black T indicator, ammonia solution, EDTA solution, Distilled water
√Chemical: sequestering agent
Procedure:
●Weigh 50ml of water sample to be tested
●Add 2.5ml of Ammonia buffer
●Add 2-3 drops of Erichrome Black T, color of the solution will be red purple
●Add required amount of sequestering agent according to dosing.
●Titrate with 0.01N EDTA sol'n, up to blue color end point.

Calculation:
*Total Hardness = (vol. of titer in ml) *(10)*

Result evaluation:

Name of sequestering agent	*Type of sequestering agent*	*Supply water hardness(ppm)*	*Hardness(ppm) reduction on dosing of 0.5 g/l sequestering agent*	*Hardness(ppm) reduction on dosing of 1 g/l sequestering agent*	*Hardness(ppm) reduction on dosing of 1.5 g/l sequestering agent*
Sample 1	*Organic phosphorous compound*	*108*	*85*	*108*	----
Sample 2	*Combination of Organic phosphorous compound & carboxylic acid compound*	*108*	*15*	*32*	*50*

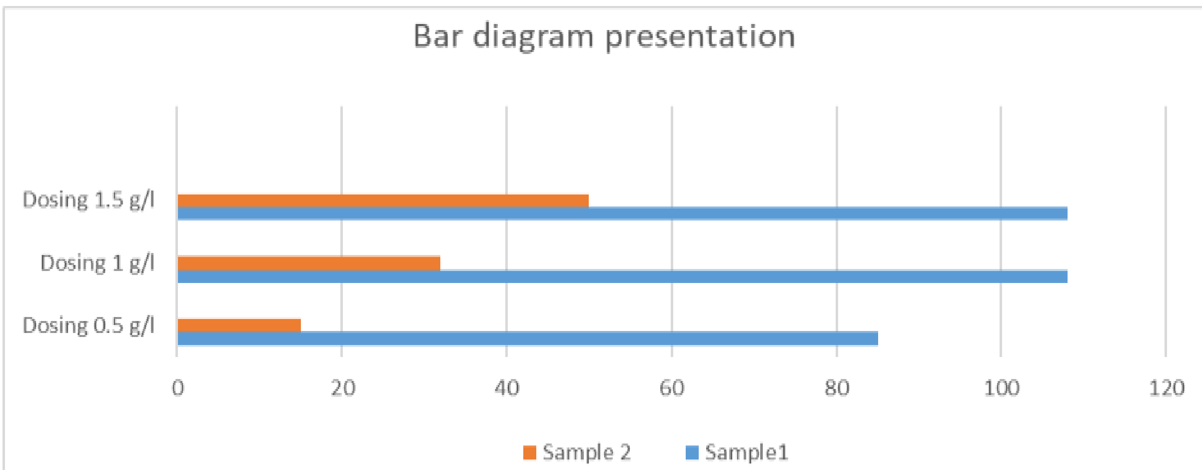

Figure: Effect of Sequestering Agent on reduction of water Hardness

From the above experimental data it is very clear to understand that with the increase of Sequestering agent dosing the water correction rates increasing significantly. For sample 1 it is seen that in 1 g/l dosing water becomes totally soft so, no need of extra dosing.

Conclusion*: So from the above discussion we have come to know that sequestering/chelating agents plays a vital role in textile wet processing. Before selecting a sequestering/chelating agents we have to know the demand of the processing condition. By selecting proper product one can avoid so many processing difficulties.*

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