Mid Semester Project report

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Xanthate End Temperature Variation

Solid Waste Viscose Management

Abstract

The increase in the exit temperature of the sodium xanthate churn is responsible for the degradation of sodium xanthate. It causes weak tyre cord fibers and a significant decrease in its elasticity and tensile strength. The exit temperature set point is between 39°C-41°C. Churns 1, 2, 3, 4, 5, 7 and 8 belong to the tyre cord section and churns 6, 9, 10, 11 and 12 belong to the CSY section. Since the churns of tyre cord section and CSY section are supplied water with independent Plate Heat Exchangers (PHE) and getting a uniform flow rate, there is a need for investigating the reason for varying exiting temperature of various churns.

Xanthation reaction takes place in an evacuated environment. Viscose on coming in contact with oxygen and other gases starts to degrade and solidifies after a few hours of exposure. The solid mass formed is useless and difficult to dispose.

The report analyses the phenomena of variation of the exit Xanthate temperature in different churns and also possible ways to dispose solid waste viscose.

Acknowledgements

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Our heartfelt appreciation goes to **Ms. Neetu Yadav** for being a competent and highly supportive instructor for Practice School-I.

About the Company

The company foundation was laid in 1952 by Syt. B K Birla; the manufacturing plant was installed in 1954 (in association with Kohorn of USA); the factory commenced production in 1956. Initially the production amounted to 5.5ton/day of Pot-Spun Viscose filament yarn, and currently produces 47 ton/day. The company installed a tyre cord unit in 1963 (in collaboration with Algemene Kuntzidjeune NV of Holland and Glanzstoff of Germany) which currently produces 18 ton/day yarn. The Century Chemical Plant was setup in 1964 to manufacture necessary raw materials like caustic, chlorine, hydrochloric acid etc., in addition to the units (from 1960) that produce carbon disulfide, and sulfuric acid. In 1998, century rayon expanded into the production of the more advanced Continuous Spun Yarn (with the help of M/s Snia Engineering of Italy) with a 10 ton/day (present) output. Century Rayon envisions to be the market leader in business of viscose filament yarn through continuous improvement in cost, technology, and quality and customer service; to care for environment, employees and society.

Century rayon is India's largest and Asia's third largest rayon producer. Its domestic market includes customers in Amritsar, Bangalore, Delhi, Surat, Panipat, etc. Its international market consists of exports in countries like South America, Europe, Japan, etc.

The products that century textile produces are: Centrifugal Pot Spun Yarn, Continuous Viscose Filament Yarn, High Tenacity Rayon Tyre Yarn. Industrial chemicals produced are Sulphuric acid, Carbon Di Sulphide and liquid chlorine produced by the auxiliary plant.

Introduction

The Tyre Cord Plant of Century Rayon produces tyre yarn and continuous spun yarn (CSY) with

a capacity of 18tons/ day ranging from 1100 to 3300den. Yarn produced is either pre-twisted or
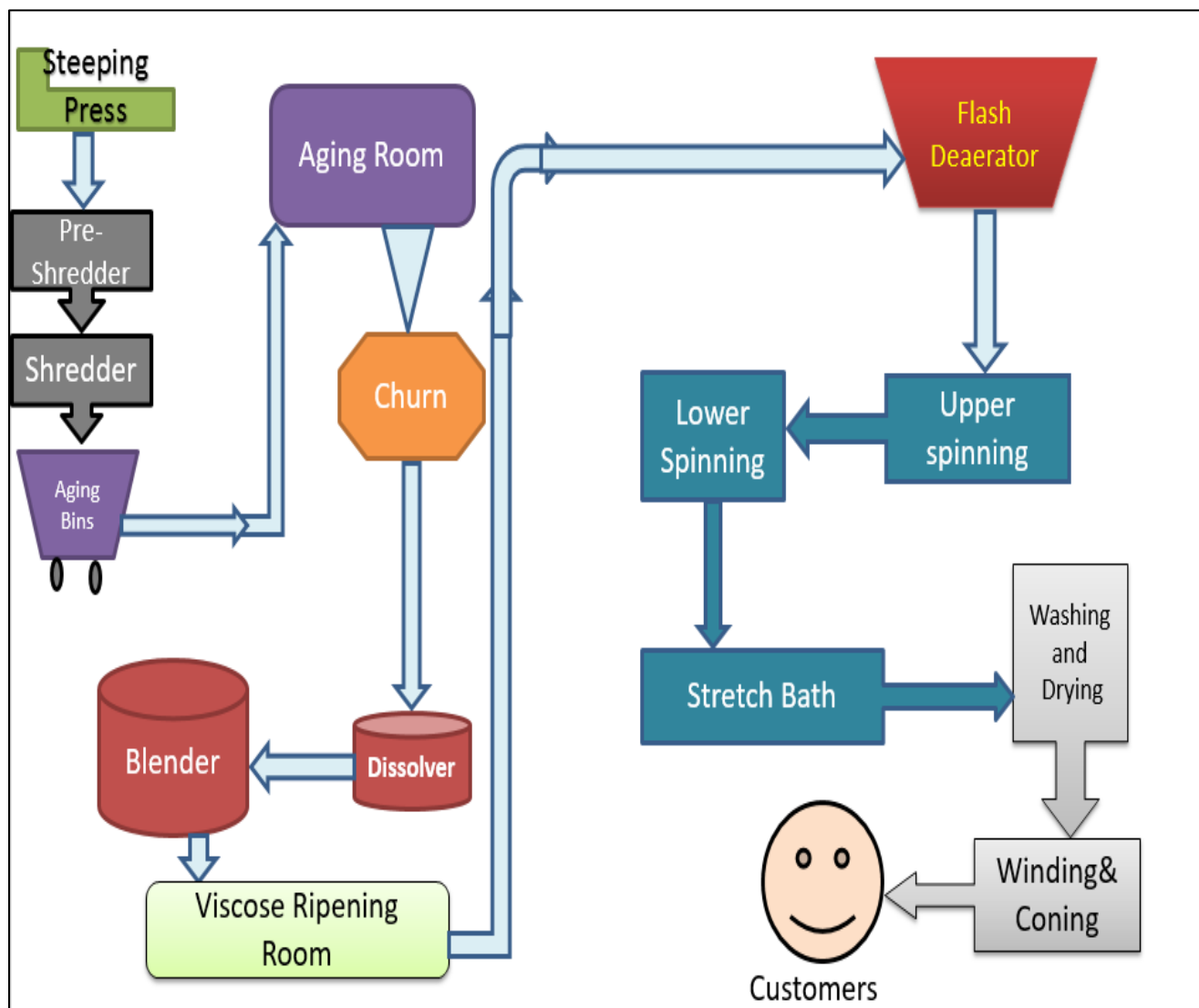
Zero Twist depending on the Customer requirements.

Figure 1. The flow diagram gives an overview of the various processes of the tyre cord plant.

Analysis of xanthate exit temperature variation in churn section of tyre cord unit

AIM:

- To study the heat transfer dynamics of the jacketed cooling in the churn section

- To observe and establish existence of a variation in exit temperature of xanthate from each churn

- To hypothesize as to the probable causes for the established variation

- To suggest, if possible, any suitable changes in design and layout of the churn section.

OBSERVATION:

- During summer, churn 7 and 8 were observed to deviate most from the desired exit temperature range of 39°C-41°C.

HYPOTHESES:

- Variations in shredder end temperature (24.5°C-27°C) can lead to temperature variations in the churn output.

- Variations in the temperature of alkali cellulose from ageing room (SP: 27°C) can lead to temperature variations in the churn output.

- Variations in CS_2 end temperature (25°C-26°C) can lead to temperature variations in the churn output.

- Variations in coolant flow in inlet to each churn can lead to temperature variations in the churn output.

- Heat transfer between coolant and surroundings during flow in pipes.

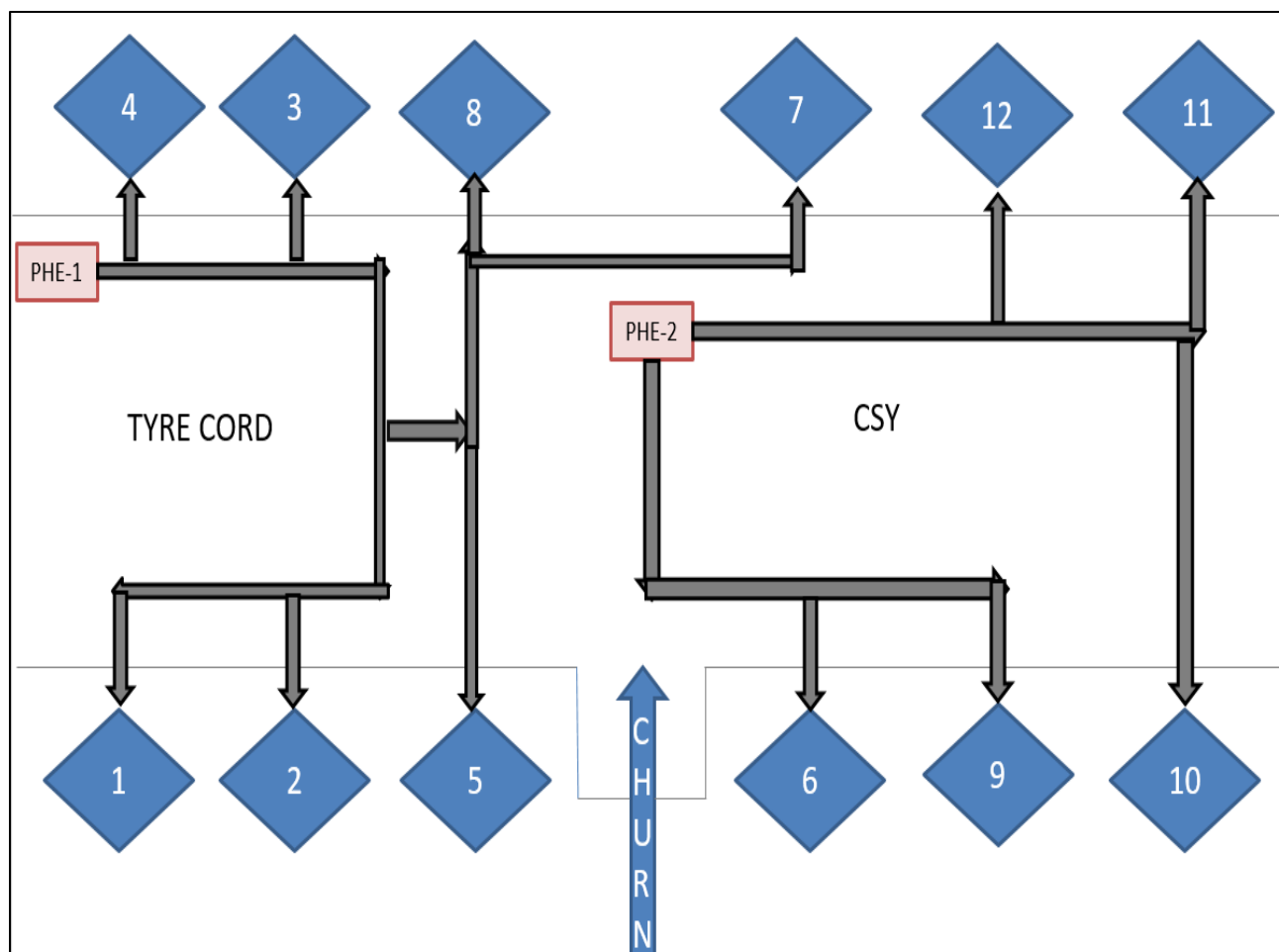- Ineffective heat transfer in jackets caused by sludge deposition, formation of dead spots, etc.

Figure 2. Piping outline of the churn cooling water.

To observe the temperature variation of churn cooling water for various churns, CCW in, CCW out, and churn temperature were noted with a five minutes interval and appropriate graphs were plotted for temperature variation with respect to churn operation time.
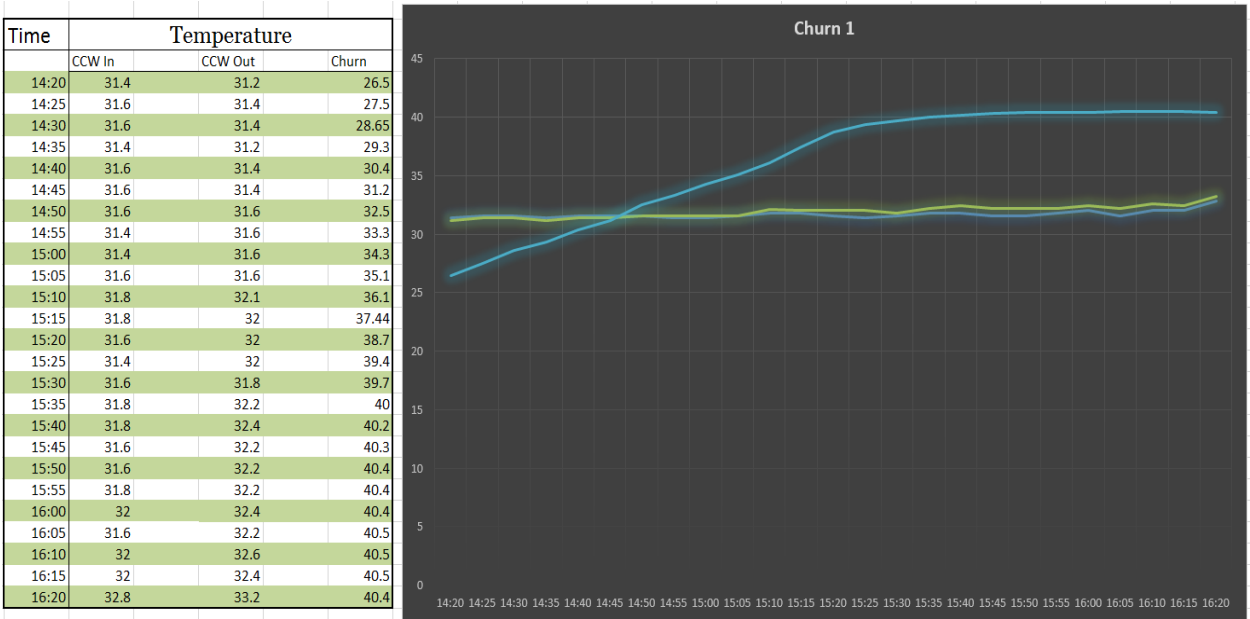
Time	Temperature		
	CCW In	CCW Out	Churn
14:20	31.4	31.2	26.5
14:25	31.6	31.4	27.5
14:30	31.6	31.4	28.65
14:35	31.4	31.2	29.3
14:40	31.6	31.4	30.4
14:45	31.6	31.4	31.2
14:50	31.6	31.6	32.5
14:55	31.4	31.6	33.3
15:00	31.4	31.6	34.3
15:05	31.6	31.6	35.1
15:10	31.8	32.1	36.1
15:15	31.8	32	37.44
15:20	31.6	32	38.7
15:25	31.4	32	39.4
15:30	31.6	31.8	39.7
15:35	31.8	32.2	40
15:40	31.8	32.4	40.2
15:45	31.6	32.2	40.3
15:50	31.6	32.2	40.4
15:55	31.8	32.2	40.4
16:00	32	32.4	40.4
16:05	31.6	32.2	40.5
16:10	32	32.6	40.5
16:15	32	32.4	40.5
16:20	32.8	33.2	40.4

Figure 3a Temperature variation with time for churn 1.

Time	Temperature		
	CCW In	CCW Out	Churn
13:25	31.5	31.6	29.3
13:30	31.4	31.5	28.2
13:35	31.4	31.4	27.5
13:40	31.6	31.6	27.1
13:55	31.4	31.2	28.5
14:00	31.4	31.4	29.7
14:05	31.4	31.4	30.6
14:10	31.4	31.4	31.7
14:15	31.4	31.6	32.8
14:20	31.4	31.6	33.6
14:25	31.4	31.8	34.5
14:30	31.2	31.6	35.7
14:35	31.2	31.6	36.9
14:40	31.2	31.6	37.7
14:45	31.4	31.8	38.3
14:50	31.6	31.8	38.7
14:55	31.4	31.8	38.9
15:00	31.4	31.8	39
15:05	31.4	31.8	39.1
15:10	31.6	32.2	39.1
15:15	31.6	32.2	39.2
15:20	31.6	32.2	39.2
15:25	31.4	31.8	39.2
15:30	31.4	31.8	39.3
15:35	31.4	32.2	39.2

Figure 3b Temperature variation with time for churn 4.

Time	Temperature		
	CCW In	CCW Out	Churn
11:20	32	31.8	27.5
11:25	31.8	31.4	31
11:30	32	31.4	30
11:35	31.6	31.6	31.8
11:40	31.4	31.6	32.5
11:45	31.4	32	34
11:50	31.6	31.8	34.9
11:55	31.8	32	35.2
12:00	31.6	32.2	36.1
12:05	31.4	31.8	38.5
12:10	31.6	31.8	38.5
12:15	31.6	31.8	39.2
12:20	31.8	32.2	40
12:25	31.6	32	40
12:30	31.6	32	40.3
12:35	31.6	32.2	40.6
12:40	31.6	32.2	40.6
12:45	31.4	32	40.6
12:50	31.4	32.2	40.8
12:55	31.5	31.8	41
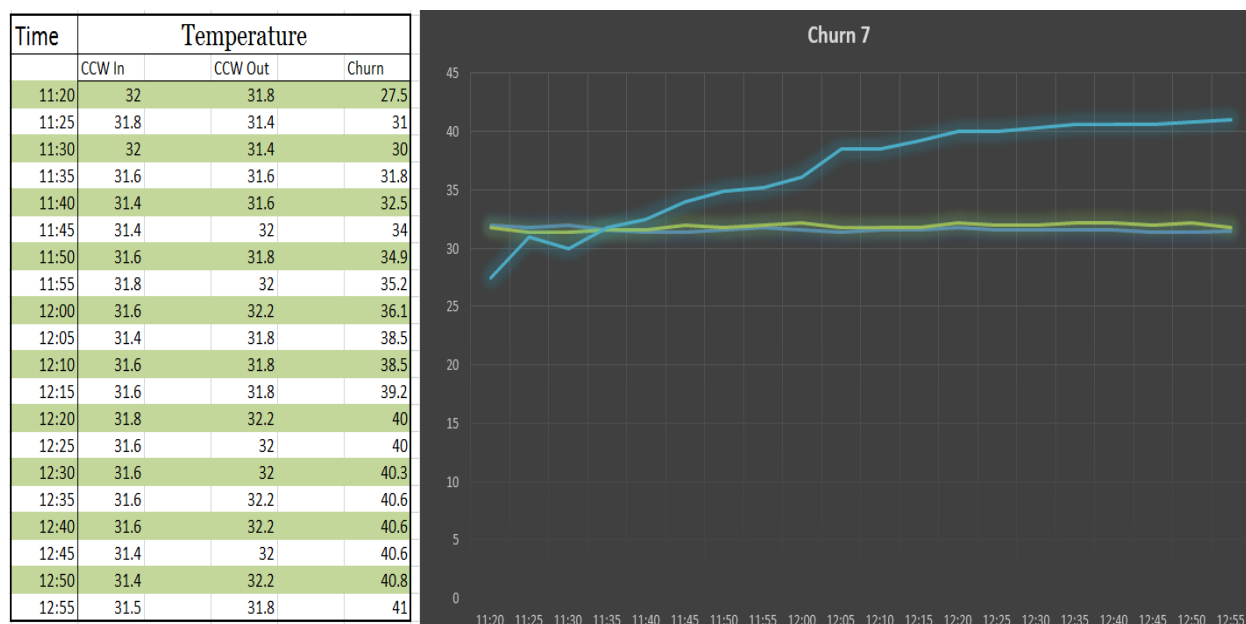
Figure 3c Temperature variation with time for churn 7.

Time	Temperature		
	CCW In	CCW Out	Churn
12:45	31.6	31.2	27.5
12:50	31.4	31.2	28.1
12:55	31.4	31.2	29.5
13:00	31.6	31.2	30
13:05	31.4	31.4	31
13:10	31.5	31.4	32.1
13:15	31.6	31.8	33.2
13:20	31.6	31.8	34
13:25	31.6	32	35.5
13:30	31.4	31.8	37.5
13:35	31.6	32	38.5
13:40	31.6	31.8	39
13:55	31.4	31.6	40
14:00	31.4	31.6	40
14:05	31.6	32	40.5
14:10	31.4	31.8	41
14:15	31.4	31.8	41
14:20	31.4	31.8	41
14:25	31.6	32	41.1
14:30	31.4	32	41
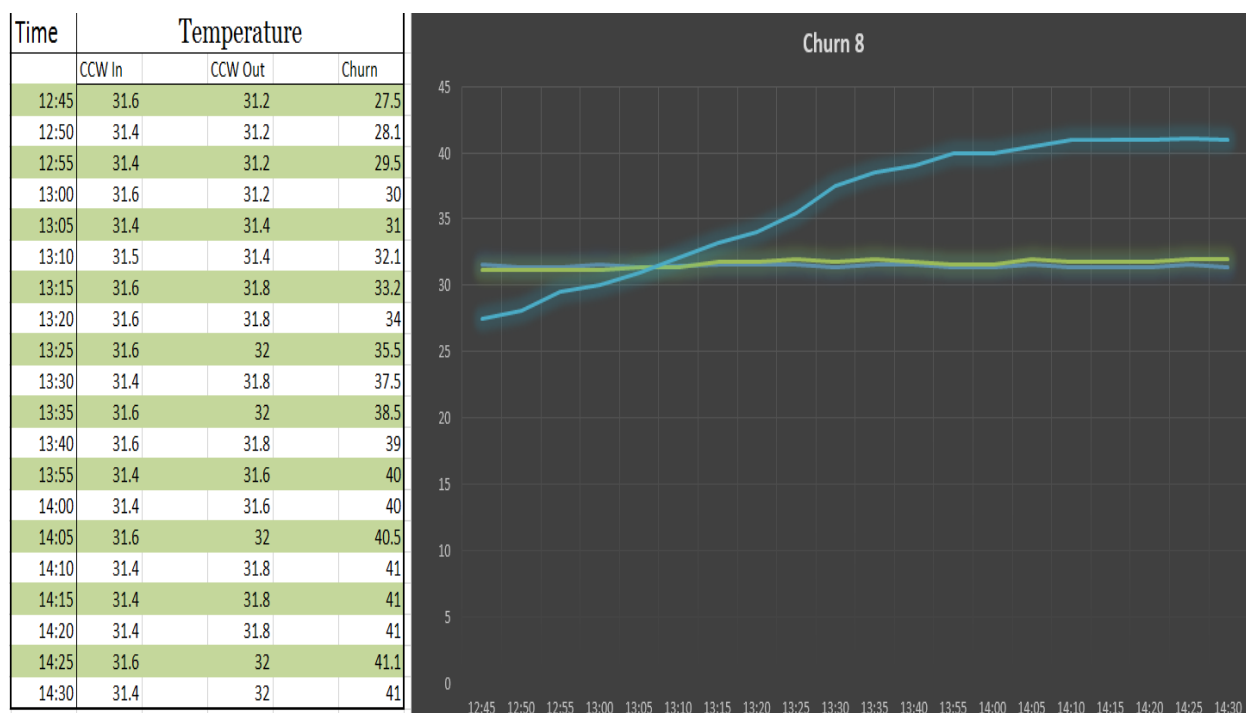
Figure 3d Temperature variation with time for churn 8.

Overall Heat Transfer Coefficient

The overall heat transfer coefficient U is a measure if the overall ability of a series of conductive

and convective barriers to heat transfer. It is commonly applied to the calculation of heat transfer

in heat exchangers, but can be applied equally well to other problems. For the case of a heat

exchanger, U can be used to determine the total heat transfer between the two streams in the heat

exchanger by the following relationship:

$$Q = UA\Delta T_{LM}$$

Where:

      Q = heat transfer rate (W)
      U = overall heat transfer coefficient (W/m²·K)
      A = available heat transfer surface area (m²)
      ΔT_{LM} = logarithmic mean temperature difference (K).

The overall heat transfer coefficient takes into account the individual heat transfer coefficients of

each stream and the resistance of the pipe material. It can be calculated as the reciprocal of the

sum of a series of thermal resistances (but more complex relationships exist, for example when

heat transfer takes place by different routes in parallel).
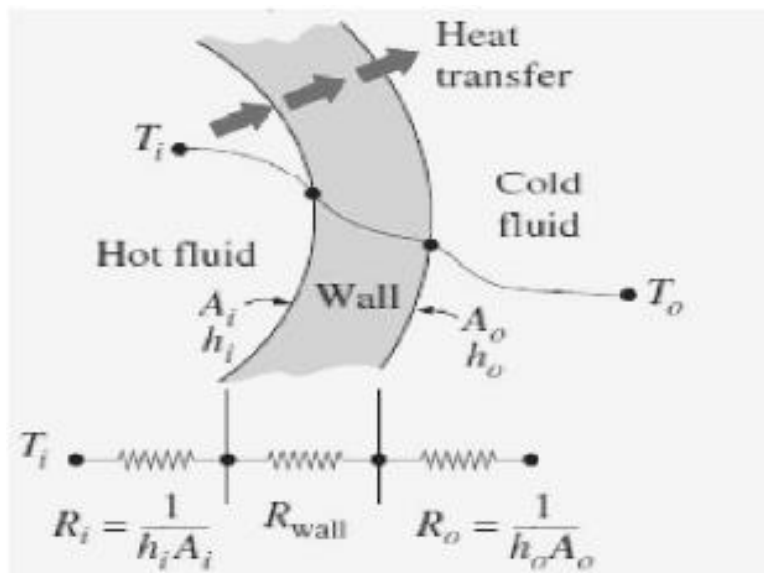
The heat transfer coefficient is the heat transferred per unit area per kelvin. Thus *area* is included in the equation as it represents the area over which the transfer of heat takes place. The areas for each flow will be different as they represent the contact area for each fluid side.

Thermal resistance due to fouling deposits:

Often during their use, heat exchangers collect a layer of fouling on the surface which, in addition to potentially contaminating a stream, reduces the effectiveness of heat exchangers. In a fouled heat exchanger the buildup on the walls creates an additional layer of materials that heat must flow through. Due to this new layer, there is additional resistance within the heat exchanger and thus the overall heat transfer coefficient of the exchanger is reduced.

Calculations

The calculations are made for the heat transfer in the jacket side and vessel side. Different equations are used for calculation purpose depending upon the assumptions taken.

Assumptions:

- There is negligible heat loss form the churn and all heat is transferred to churn cooling water

- The average temperature of each churn is assumed to be constant for calculation purpose

- Properties of CS_2 are considered for vessel side calculations

- Due to low rpm of churn (2rpm), it is safe to assume it as a stationary vessel

- No fouling is considered while making heat transfer calculations

Equations for jacket side HE and vessel side HE are as follows:

Jacket side heat transfer equations:

$$\text{Nu} = \frac{\alpha_s d_e}{\lambda},$$

$$\text{Re} = \frac{d_e \rho (\sqrt{v_i v_A} + v_B)}{\eta},$$

$$d_e = 0.816 (D_j - D_T),$$

$$v_i = \frac{4\dot{V}}{\pi d_i^2}, \quad v_B = 0.5 \sqrt{2z\beta g \Delta T_s}$$

$$\text{Nu} = \frac{0.03 \text{Re}^{3/4} \text{Pr}}{1 + 1.74 \text{Re}^{-1/8} (\text{Pr} - 1)} \left(\frac{\eta}{\eta_w} \right)^{0.14}$$

$$\text{Radial}; v_A = \frac{4\dot{V}}{\pi (D_j^2 - D_T^2)}, \quad \text{Tangential}; v_A = \frac{2\dot{V}}{(D_j - D_T)z}$$

Vessel side heat transfer equations:

$$\text{Nu}_D = 0.47 (\text{Gr}_D \text{Pr})^{1/4}$$

$$\text{Gr}_D = g\beta D^3 \Delta T / v^2$$

Calculation of overall heat transfer coefficient for the entire vessel is made as follows:

$$U_i = \frac{1}{\frac{1}{h_i} + \frac{A_i \ln (r_o/r_i)}{2\pi kL} + \frac{A_i}{A_o} \frac{1}{h_o}}$$

$$U_o = \frac{1}{\frac{A_o}{A_i} \frac{1}{h_i} + \frac{A_o \ln (r_o/r_i)}{2\pi kL} + \frac{1}{h_o}}$$

$$q = UA \, \Delta T_{\text{overall}}$$

$$q = \frac{T_A - T_B}{\frac{1}{h_i A_i} + \frac{\ln (r_o/r_i)}{2\pi kL} + \frac{1}{h_o A_o}}$$

Churn Heat Transfer Calculations

DATA

Water flow rate calculations

Pump Capacity	60	m^3/hr	Volumetric flow rate in header	0.016666667	m^3/s
Header piping diameter(4")	0.1016	m	Header piping area	0.008107313	m^2
Tapping piping diameter(2")	0.0508	m	Tapping piping area	0.002026828	m^2
No of splits	7		Volumetric flow rate in tapping	0.002380952	m^3/s
Velocity of water in tapping v_i	4.70125	m/s	Velocity of water in header	2.055757196	m/s

Properties of water

Dynamic Viscosity μ	0.000764	Pa.s	Density ρ	995	kg/m^3
Thermal Conductivity K	0.061842	W/m°C	Specific heat C_p	4.178	kJ/kg.K
Prandtl No Pr	5.2		Specific Volume v	1.005	m^3/kg
Expansion Coefficient β	0.000321	K^{-1}	Heat of Vaporization H_v	2426	kJ/kg

Properties of CS_2

Dynamic Viscosity μ	0.000421	Pa.s	Density ρ	1256	kg/m^3
Thermal Conductivity K	1.56623	W/m°C	Specific heat C_p	1.039	kJ/kg.K
Prandtl No Pr	0.865		Specific Volume v		m^3/kg
Expansion Coefficient β	0.2264	K^{-1}	Heat of Vaporization H_v		kJ/kg

Properties of Mild Steel

Thermal Conductivity K	31.5	W/m°C	Density ρ	7850	kg/m^3

Churn characteristics

Vessel diameter D_T	1.28	m	Vessel area A_i	14.63729613	m^2
Jacket diameter D_j	1.4	m	Jacketed vessel area A_o	16.271878	m^2
Length L	3	m			

JACKET SIDE CALCULATION

$$Nu = \frac{\alpha_s d_e}{\lambda},$$

$$Re = \frac{d_e \rho (\sqrt{v_i v_A} + v_B)}{\eta},$$

$$d_e = 0.816(D_j - D_T),$$

$$Nu = \frac{0.03 \, Re^{3/4} \, Pr}{1 + 1.74 Re^{-1/8}(Pr - 1)} \left(\frac{\eta}{\eta_w}\right)^{0.14}$$

$$v_i = \frac{4\dot{V}}{\pi d_i^2}, \quad v_B = 0.5\sqrt{2z\beta g \Delta T_s}$$

$$Radial; v_A = \frac{4\dot{V}}{\pi(D_j^2 - D_T^2)},$$

d_e	0.09792	m	K	0.61842	W/m°C
g	9.81	m/s^2	Pr	5.2	
μ	0.000764	Pa.s			
β	0.000321	K^{-1}			
ρ	995	kg/m^3			
v_i	4.70125	m/s	sqrt(v_i*v_A)	0.21051316	m/s

v_A	0.00943	m/s	R_e	26846.0489	
Nu	107.5317105		$h_i=Nu*K/d_e$	679.12337	W/m²°C

VESSEL SIDE CALCULATION

$$Nu_D = 0.47(Gr_D Pr)^{1/4}$$

$$Gr_D = g\beta D^3 \Delta T/v^2$$

Pr	0.865		K	1.56623	W/m°C
g	9.81	m/s²	ΔT	13.5	K
μ	0.00042	Pa.s	v	3.3519E-07	
β	0.2264	K⁻¹	D_T	1.28	m
ρ	1256	kg/m³			
GrD	5.5966E+11		NuD	392.0423045	
$h_o=Nu*K/D_T$	479.71	W/m²°C			

VESSEL THERMAL RESISTANCE

$$R=\ln(D_T/D_j)/2\pi KL$$

R	0.00015	W/m°C

OVERALL HEAT TRANSFER COEFFICIENT CALCULATION

hi	679.1233699	ho	392.0423045
1/hi	0.001472487	1/ho	0.002550745
Ai/Ao	0.899545592	Ai/Ao*1/ho	0.002294512
1/Ui		0.005976104	
Ui		27.81641168	

CHURN AVAILABLE AREA CALCULATION

Assumption: We are maintaining constant average churn temperature

CHURN #1							Water flow rate	2.36905	kg/s
ΔT_m	4.02844	K	Availablle churn area	24.27538109	m^2		Heat gained by water	2720.22	W
CHURN #4							Water flow rate	2.36905	kg/s
ΔT_m	3.7735	K	Availablle churn area	27.17049413	m^2		Heat gained by water	2851.957	W
CHURN #7							Water flow rate	2.36905	kg/s
ΔT_m	4.4111	K	Availablle churn area	22.19395593	m^2		Heat gained by water	2723.22	W
CHURN #8							Water flow rate	2.36905	kg/s
ΔT_m	3.9868	K	Availablle churn area	15.62654628	m^2		Heat gained by water	1732.96	W

- Since the area available for churn #7 and #8 is less compared to churns #1 and #4, the temperature of the xanthate exiting from them is greater.
- Proper flushing of the jackets can be done to avoid this problem.

Observations

It is observed that the actual heat transfer available for each churn is $26m^2$. Theoretically the available area for each churn is shown above.

- Churn area available for #1 and #2 is more compared to churns 7 and 8

- There is better heat transfer occurring in churns 1 and 4 compared to 7 and 8 due to more area available

Conclusions

Better flushing of jackets can be done to avoid formation of dead spots.

Also rust formation can be avoided by taking care of jacket material.

Efficient jackets can be used by using dimpled jackets or jackets with cooling helical coil for efficient heat exchange.
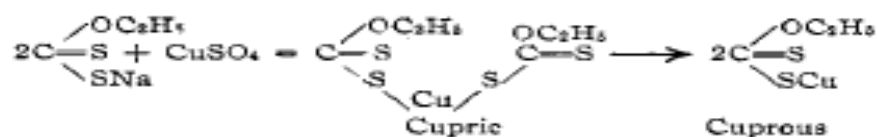
Solid viscose waste management

AIM:

- To study the existing disposal methods of waste viscose, solid and liquid form

- To suggest reagents to recover cellulose from solid viscose

- To suggest reagents to safely dispose viscose waste

OBSERVATION:

- Cellulose can be recovered from waste liquid viscose by treating it with 10% HCl

  solution

- Solid waste viscose can be used for land-filling, but the caustic seepage during monsoon

  is a health hazard.

Reactions

1. Reactions with cupric salts to give a brown precipitate

$$2C\underset{\diagdown SNa}{\overset{\diagup OC_2H_5}{=}}S + CuSO_4 = C\underset{\diagdown S}{\overset{\diagup OC_2H_5}{=}}S \quad \underset{S}{\overset{OC_2H_5}{=}}C\underset{Cu}{=}S \longrightarrow 2C\underset{\diagdown SCu}{\overset{\diagup OC_2H_5}{=}}S$$

Cupric   Cuprous

Solutions of salts of heavy metals precipitate metallic xan-
thates and regenerate cellulose.

2. Action of alkyl iodides to form esters

(2) *Action of alkyl iodides on the salts to form esters.*

$$C\underset{\diagdown SNa}{\overset{\diagup OR}{=}}S + IC_2H_5 = C\underset{\diagdown SC_2H_5}{\overset{\diagup OR}{=}}S + NaI$$
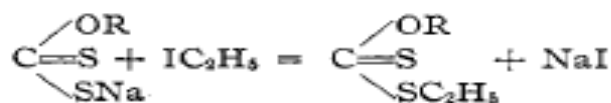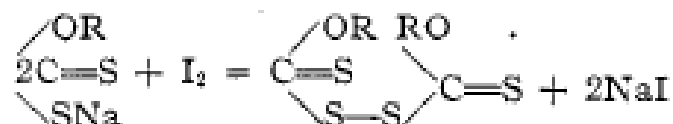
These are liquids possessing an odor of garlic and insoluble
in water. Ammonia decomposes them into mercaptans and
esters of sulfocarbamic acid—i. e., thiourethanes.

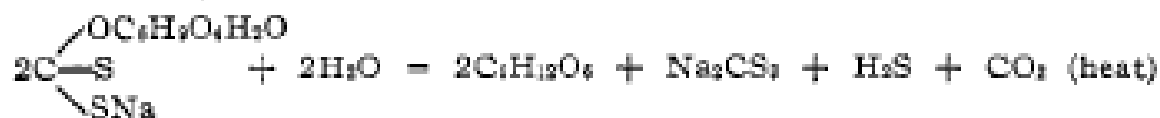3. Action of halogenated fatty acids:

$$\underset{\underset{\diagdown SNa}{C=S}}{\overset{\diagup OR}{}} + ClCH_2COOH = \underset{\underset{\diagdown SCH_2COOH}{C=S}}{\overset{\diagup OR}{}} + NaCl$$

Ethyl xanthic acetic acid

4. Action of iodine:

$$2\underset{\underset{\diagdown SNa}{C=S}}{\overset{\diagup OR}{}} + I_2 = \underset{\underset{\diagdown S-S}{C=S}}{\overset{\diagup OR}{}} \underset{\diagdown}{\overset{RO}{}} C=S + 2NaI$$
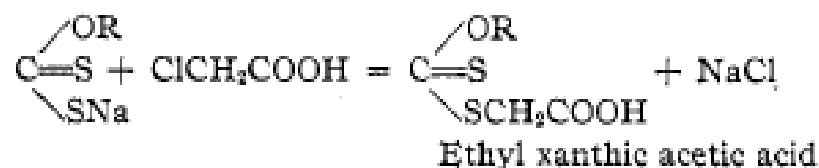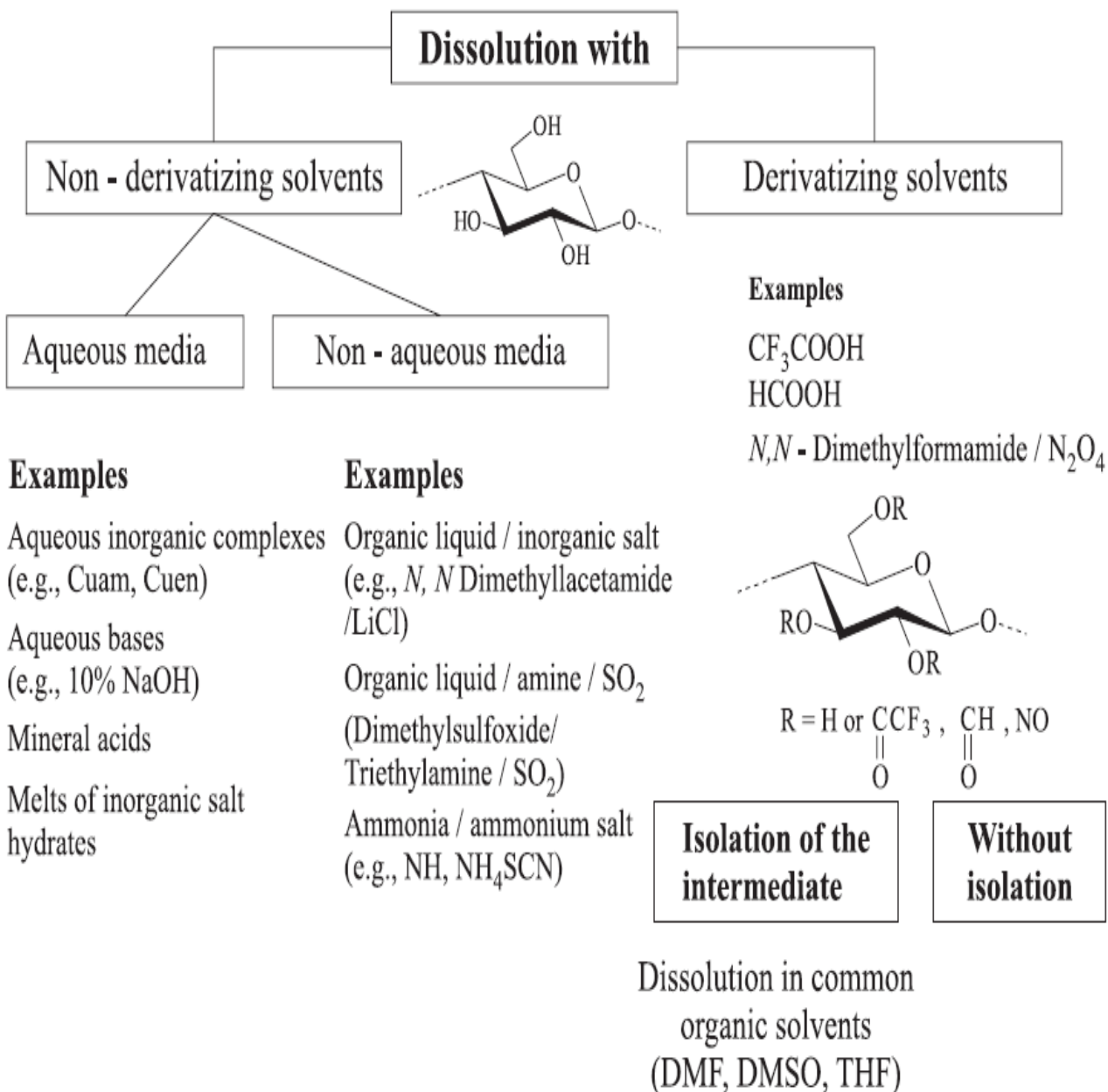
The next important step after reaction of viscose is purification of formed products to remove

hazardous chemicals formed.

Purification

Cellulose xanthate or its solution may be purified by means of water, heat, dialysis, acids, excess alkali, alum, sodium carbonate, carbon dioxide, sulfur dioxide, acid sodium sulfite, etc. Some of these methods may be represented by the following equations:

$$\underset{\underset{\diagdown SNa}{C-S}}{\overset{\diagup OC_6H_9O_4NaOH}{}} + H_2O = \underset{\underset{\diagdown SNa}{C-S}}{\overset{\diagup OC_6H_9O_4H_2O}{}} + NaOH \text{ (water)}$$

$$2\underset{\underset{\diagdown SNa}{C-S}}{\overset{\diagup OC_6H_9O_4H_2O}{}} + 2H_2O = 2C_6H_{12}O_6 + Na_2CS_3 + H_2S + CO_2 \text{ (heat)}$$

$$3CS_2 + 6\,NaOH = Na_2CO_3 + 2Na_2CS_3 + 3H_2O \text{ (excess caustic)}$$

Reagents for regeneration of cellulose:

Dissolution with

Non - derivatizing solvents

Derivatizing solvents

Aqueous media

Non - aqueous media

Examples

CF_3COOH
HCOOH

N,N - Dimethylformamide / N_2O_4

$R = H$ or CCF_3, CH, NO

Examples

Aqueous inorganic complexes
(e.g., Cuam, Cuen)

Aqueous bases
(e.g., 10% NaOH)

Mineral acids

Melts of inorganic salt
hydrates

Examples

Organic liquid / inorganic salt
(e.g., N, N Dimethyllacetamide
/LiCl)

Organic liquid / amine / SO_2
(Dimethylsulfoxide/
Triethylamine / SO_2)

Ammonia / ammonium salt
(e.g., NH, NH_4SCN)

Isolation of the intermediate

Without isolation

Dissolution in common
organic solvents
(DMF, DMSO, THF)

Number of components	Substance group	Examples
Unicomponent	N-Alkylpyridinium halogenides	N-Ethylpyridinium chloride
	Oxides of tertiary amines	N-Methylmorpholine-N-oxide
		Triethylamine-N-oxide
		N-Methylpiperidine-N-oxide
Bicomponent	Dimethyl sulfoxide (DMSO) containing solvents	DMSO/methylamine
		DMSO/KSCN
		DMSO/CaCl$_2$
		DMSO/TBAF
	Liquid ammonia/ sodium or ammonium salts	NH$_3$/NaI (NH$_4$I) NH$_3$/NaSCN (NH$_4$SCN)
	Dipolar aprotic solvents/LiCl	N,N-Dimethylacetamide/LiCl N-Methylpyrrolidone/LiCl
	Pyridine or quinoline containing systems	Pyridine/resorcinol Quinoline/Ca(SCN)$_2$
Tricomponent	NH$_3$ or amine/ salt/polar solvent	NH$_3$/NaCl/DMSO
		Ethylenediamine/NaI/
		N,N-Dimethylformamide
	NH$_3$ or amine/SO$_2$ or SOCl$_2$/polar solvent	Diethylamine/SO$_2$/DMSO

a) In most cases a preactivation of the cellulose is required.

Reason for choosing DMSO as a reagent:

The solvent DMSO/TBAF is a more efficient reaction medium for transesterification reactions than NMNO. Conversions of cellulose with vinyl acetate as acylating reagent yielded cellulose acetates with DS values up to
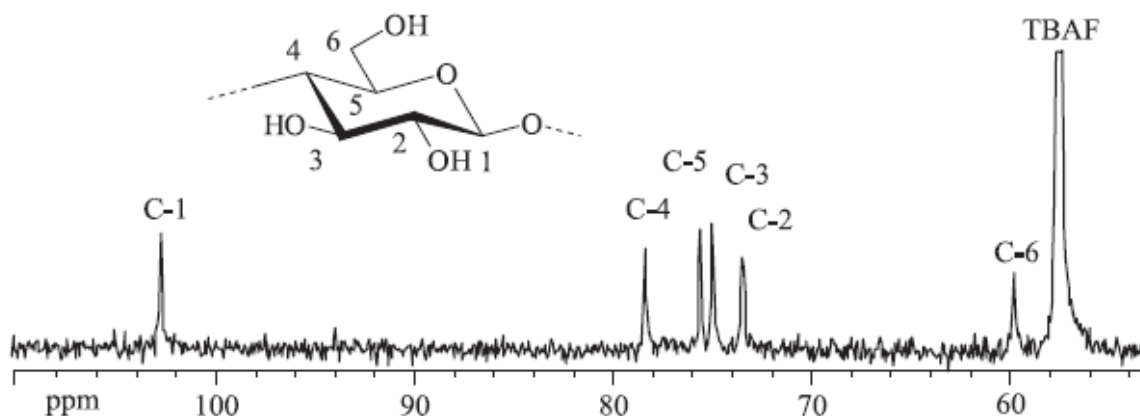

Figure 2. ^{13}C-NMR spectrum of cellulose (3%, w/v) dissolved in dimethyl sulfoxide-d_6/tetrabutylammonium fluoride trihydrate (TBAF).

Roadmap towards completion of the experiment:

- Since DMSO is a selective organic solvent that dissolves cellulose, experiments need to be done to check whether it reacts with solid viscose

- One important reaction to be considered is reaction of viscose with DMSO and paraformaldehyde to form hemiacetal. Hemiacetal hydrolyzes to cellulose on addition of water

- The viscose removed from gauge glass can be immediately dissolved in water to prevent the solidification of viscose and can be stored for a longer period of time in order to react with aforementioned reagents

References

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