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Physicochemical Characteristics of Rayon Grade Dissolving Pulp and the Effects of Metallic-Ions on the Viscose Rayon Process

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Abstract. Pakistan imports rayon grade pulp from different countries for viscose rayon fibre manufacturing. Samples of imported pulp were collected and analyzed for alpha-cellulose, hemicellulose, calcium, magnesium, silica, copper, manganese, and iron. Moisture, ash content, cuprammonium viscosity, degree of polymerization, alkali absorption, and colour brightness were also determined. The results showed that all these parameters varied from sample to sample. The cotton linter pulp contained high alpha-cellulose content (94-98%) as compared to the softwood pulp (89.7-95%). Degree of polymerization of all samples was above 500 and varied from 500-750 ml/g. The study showed that higher manganese and copper content in cotton decreased the degree of polymerization. Iron above the standard value (7-10 ppm) affected the brightness of fibre, as observed in the case of cotton linter pulp (imported from China). The percentage of ash was less than 0.25% in all the samples studied.

Keywords: rayon pulp, pulp characteristics, metallic ion effects, viscose, rayon fibre, viscose process, dissolving pulp

Introduction

The development and use of a great variety of man-made fibres have created a revolution in textile industry in the recent decades. Rayon is the commercial man-made fibre composed of regenerated cellulose. Pulp is chiefly prepared from wood and cotton linter and is used for the manufacturing of viscose rayon fibre (Shareve, 1977). The term 'dissolving pulp' is used for pulp dissolved in the form of alkali soluble cellulose xanthate in the viscose process. Bamboo is being used at present for the manufacture of dissolving pulp. The alpha-cellulose content of the dissolving pulp is important because the rayon yield depends upon it. If it is low, there will be difficulties in processing. The required alpha-cellulose content of dissolving pulp for viscose staple fibre is 89 to 93 %. Higher content of hemicellulose in the pulp spoils the caustic soda lye used for steeping in the viscose manufacture. The hemicelluloses are mostly lower polymers of pentoses and should be less than 4% for the rayon grade dissolving pulp (Mandelia, 1970). Degree of polymerization and cuprammonium viscosity of the pulp is very important for the reduction of molecular weight of the cellulose to get a viscose of right viscosity (Edwin, 1948). The polymerization degree (DP) of the hardwood dissolving pulp for the viscose rayon continuous filaments is generally 550 to 600.

An important quality index for the dissolving pulp is the filterability of viscose made from it. Before spinning, the viscose has to be filtered to avoid the plugging of the spinnerets. Although slow viscose filtration may be due to many causes, the pulp properties influence the viscose filterability to a large extent. Chlorine dioxide helps in increasing the pulp brightness and minimizing the colour reversion. Sulphur dioxide helps in reducing the ash content (Mandelia, 1970). Alkalis are able to swell cellulose at low concentrations and produce dispersion at high concentrations. Certainly, the alkali opens up the fibre structure and leads to enhanced reactivity of the pulp during the xanthation process.

The dissolving pulp, especially for viscose, must be highly purified and uniform in quality (Edwin, 1948). The ash and its constituents are very important in deciding the suitability of the pulp for rayon. Calcium and magnesium are usually found in pulps prepared by the sulphite process. Metallic ions give trouble in the manufacture of viscose and in its filtration (Mandelia, 1970).

The viscose industries convert the dissolving cellulose pulp into products such as staple fibre, cards, films, packing materials, and non-edible sausage casings.The materials are used in the clothing, drapery, hygiene, automobile, food, and packaging industries (Ewing and Stepanik, 2000).

Materials and Methods

Nine rayon grade pulp samples of cotton linter and wood pulp were collected from Chemi Viscose Fiber Pvt., Nawabshah, Sindh, Pakistan.The ash content was determined by igniting the sample of pulp at 580-600 °C in a muffle furnace (ASTM D-586-97, 2002), while silica was determined by heating the sample at 900 °C. The moisture content was analyzed at 105 °C in a drying oven for two h (ASTM D-644-99, 2002). Calcium and

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magnesium were determined volumetrically (Furman, 1963). Atomic absorption spectrophotometer (Hitachi , model Z-8000) was used for the determination of iron, copper and manganese (ASTM D-4085-93, 2002).

For the determination of alpha-cellulose and the hemicellulose contents, standard testing method of TAPPI was used (TAPPI-T203-cm-99, 2000). Cuprammonium disperse viscosity of 1% solution of pulps was determined by using the capillary viscometer, in cuprammonium solution, having the copper concentration of 14.8-15.2 g/l and the ammonium concentration of 190-210 g/l (Scott, 1956). The brightness of pulp samples was determined by a photovolt model-577 brightness meter (ASTM D-985-97, 2002). The small round slices of pulp samples were immersed into 60 ml 17.50% NaOH solution for the determination of alkali absorption capacity (Charles, 1950).

Results and Discussion

Table 1 shows that alpha-cellulose ranged from 89.83 to 98.12%. Sulphide dissolving hardwood pulp **(e)** had the lowest alpha-cellulose content, while cotton linter pulp **(i)** had the highest alpha-cellulose. The alpha-cellulose is that fraction which is resistant to 17.5% sodium hydroxide at 20 ˚C. The amount of alpha-cellulose in the pulp should be more than 89%.

Hemicellulose is the principal non-cellulosic polysaccharide present in the pulp. The presence of hemicellulose in viscose pulp is not desirable because it causes deterioration of mechanical properties, such as the wet strength of the finished fibre (Mandelia, 1970). Hardwood contains more hemicellulose as compared to softwood (Table 1).

Moisture content showed little variation, as compared to other parameters. Moisture is also very important in the pulp as the sorption of water affects the degree of swelling of the pulp. Therefore, the moisture content in the rayon grade pulp should not exceed more than 5%.

The percentage of ash was less than 0.25% in all the samples. Cotton linter pulp **(g)** had the highest ash content, which was 0.24%. The ash and its constituents are very important in deciding the suitability of the pulp for rayon. It should be under 0.1% of the weight of the pulp. The pulp ash contents mostly consist of salts and hydroxides of the multivalent elements, such as silica, calcium, magnesium and iron, which are not desirable in the pulp because they create troubles in the filtration of the viscose.

The degree of polymerization of all samples was above 500 and varied from 500-750. The cupprammonium viscosity of the pulp was directly proportional to the degree of polymerization. The degree of polymerization is necessary for regulating the viscosity of the viscose for spinning. The degree of polymerization of cellulose must be between 450-500, otherwise the strength, elongation, the spinning state, and dye affinity of the yarn is affected.

The brightness of most of the samples was below the standard values. The required brightness of the rayon grade pulp should be above 91% because it affects the brightness of the final product.

Many pulp properties have a significant effect on the viscose properties and subsequently the fibre properties. These include the degree of polymerization, the oxidation state of cellulose, the soda solubility, and the residual level of pulp impurities like iron and silica (Calvin, 2001). The pulp samples that contain high calcium, magnesium and silica contents in ash, may cause poor filterability after the ripening process.

Table 1. Physicochemical characteristics of the rayon grade wood pulps and cotton linter pulps

Test	Visconier-F-MP softwood (USA) **a**	Fibernier-F softwood (USA) **b**	Tembec softwood (Canada) **c**	Domjoe softwood (China) **d**	Sulphite dissolving hardwood (Canada) **e**	Hardwood (Canada) **f**	Goami cotton linter (China) **g**	Nanjiang cotton linter (China) **h**	Pulp-651 cotton linter (USA) **i**
α-Cellulose (%)	94.02	94.47	93.64	94.78	89.83	94.52	94.63	94.37	98.12
Hemicellulose (%)	3.78	4.59	7.80	5.23	4.44	3.91	5.57	5.18	1.9
Moisture (%)	5.27	6.95	6.40	5.57	5.84	6.04	5.54	5.96	4.83
Ash (%)	0.10	0.14	0.03	0.05	0.12	0.10	0.24	0.19	0.08
Depolymerization	710	727	692	510	590	518	506	715	745
Cuprammonium viscosity (centipoise)	12.80	13.0	11.40	7.50	10.41	8.70	7.70	11.80	16.30
Brightness (%)	90.5	87.3	92.7	88.6	91.0	91.10	77.40	89.10	87.40
Alkali absorption (%)	480	443	493	450	472	438	639	394	469

Table 2. Metallic ions (ppm) present in the rayon grade pulps

Test	Visconier F-MP softwood (USA)	Fibernier-F softwood (USA)	Tembec softwood (Canada)	Domjoe softwood (China)	Sulphite dissolving hardwood (Canada)	Hardwood (Canada)	Goami cotton linter (China)	Nanjiang cotton linter (China)	Pulp-651 cotton linter (USA)
	a	**b**	**c**	**d**	**e**	**f**	**g**	**h**	**i**
Calcium	31.50	17.60	23.17	68.61	75.02	82.1	128.04	105.3	13.60
Magnesium	16.09	8.99	11.49	29.84	32.57	39.50	61.50	47.0	7.88
Silica (ppm)	15.93	5.41	12.16	33.25	28.0	37.08	52.90	62.35	4.07
Copper	1.39	0.96	1.16	0.89	3.42	3.46	3.85	2.18	0.41
Iron	3.89	1.82	4.33	6.11	12.66	15.3	18.73	16.64	2.80
Manganese	0.07	0.12	0.17	0.92	1.29	1.77	1.21	1.58	0.18

The subsequent viscose ripening was combined with filtration through a metal sieve fabric to eliminate persistent fibre fragments and to reduce the gel particles content of a sufficiently low level. Good filtration of viscose was required before spinning for the production of a regular viscose rayon staple fibre. The viscose filterability characterizes the quality of a viscose solution (Sixta *et al.*, 2004).

Cotton linter **(g, h)** and hardwood pulp samples **(e, f)** contained high calcium and magnesium as given in Table 2. The metallic ions cause trouble in the manufacture of viscose, which in turn is responsible for poor filterability and chocking of spinnerets during the spinning process. Manganese, copper, and iron are very harmful if present in the ash in more than the permissible minimum. Manganese and copper accelerate the degradation of cellulose rapidly during the ageing process of alkali cellulose. In the case of cotton linter and hardwood pulp samples, both manganese and copper contents were above the permissible limit (Table 2).

The maximum permissible content of magenese in the dissolving pulp for viscose is 0.2 ppm and that of copper is 3 ppm. Iron above the standard value (7-10 ppm) affects the quality of the viscose and lowering the brightness of fibre, as was observed in the case of cotton linter pulp.

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