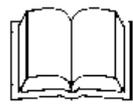


Research Group



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Association of independent consultants and experts
in field of mineral resources, metallurgy and chemical industry

Viscose Pulp Market Research in the CIS

Sample PDF

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*Internet: www.infomine.ru
e-mail: info@infomine.ru*

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Note: PPM means Pulp&Paper Mill

Annotation

The report is devoted to review of market of viscose pulp in the CIS (it should be noted that acetate pulp is not produced in the CIS at all). The report has been prepared on the basis of studying and analysis of data of Federal Service of State Statistics of Russia (FSSS of Russia), Federal Customs Service of Russia (FCS of Russia), as well as data of «InfoMine». The report is composed of 6 Chapters, contains 49 pages, including 19 Tables, 16 Figures and Appendix.

The first Chapter of the report presents data on available technologies of production of viscose pulp, their features, required resources and their quality.

The second Chapter of the report is devoted to analysis of production of viscose pulp in the CIS. The Chapter presents normative requirements, imposed on quality of viscose pulp, statistics of production of the product in 1997-2007, describes the main Russian producers of viscose pulp.

The third Chapter analyses foreign trade operations of Russian companies in viscose pulp, presents data on volumes of export and import of the products, estimates regional pattern of supplies, presents data on volumes of supplies of viscose pulp by leading Russian exporters of the product.

The fourth Chapter presents data on dynamics of export and import prices on the product for the latest 7 years.

The fifth Chapter the report presents supply-demand balance of viscose pulp in Russia in 1999-2007, describes the main Russian consumers of viscose pulp, analyses factors, which owe demand for viscose pulp.

The sixth Chapter of the report is devoted to forecast of development of production and consumption of viscose pulp in Russia up to 2012.

Introduction

Viscose pulp (also called “dissolving pulp”) is a kind of pulp, designated for chemical processing. After bleaching, artificial fiber for manufacture of silk and staple fabrics is produced from this pulp at viscose plants. Unbleached (brown) pulp, designated for bleaching for viscose pulp, should be deeply and evenly cooked, has low viscosity and homogenous composition.

Domestic mills commonly produce viscose pulp with kappa number after cooking of 40-50 and cuprammonium viscosity (1% solution) of 40-50 MPa·s.

Depending on pulping method, sulfate and sulfite viscose pulp is distinguished.

Pulp triacetate, also known simply as triacetate and acetate pulp, is manufactured from pulp and acetate. Triacetate is typically used for the creation of fibers and film base. It is similar chemically to pulp acetate, with the distinguishing characteristics being that in triacetate, according to the Federal Trade Commission definition, at least "92 percent of the hydroxyl groups are acetylated." During the manufacture of triacetate the pulp is completely acetylated whereas in regular pulp acetate or pulp diacetate, it is only partially acetylated. Triacetate is significantly more heat resistant than pulp acetate.

Triacetate was first produced commercially in the U.S. in 1954 by Celanese Corporation.

Triacetate is derived from pulp by combining pulp with acetate from acetic acid and acetic anhydride. The pulp acetate is dissolved in a mixture of dichloromethane and methanol for spinning. As the filaments emerge from a spinneret, the solvent is evaporated in warm air, in a process known as dry spinning, leaving a fiber of almost pure triacetate.

A finishing process called S-Finishing or surface saponification is sometimes applied to acetate and triacetate fabrics using a sodium hydroxide solution. This removes part or all of the acetyl groups from the surface of the fibers leaving them with a pulp coating. This reduces the tendency for the fibers to acquire a static charge.

At present time, acetate pulp is not produced in the CIS at all. Mitsubishi Rayon Co., Ltd. is the only company that still produces the fiber in the world. Eastman Kodak formerly manufactured pulp triacetate. They discontinued this product on March 15, 2007.

I. Technology of production of viscose pulp (pulping)

Chemical pulping achieves fiber separation by dissolving the lignin that cements the fibers together. There are two major chemical processes: Sulfate and Sulfite pulping.

Sulfate Pulping (the Kraft process, also known as Kraft pulping or sulfate process). The makeup chemical for sulfate pulping is essentially sodium sulfate. This process is usually referred to as the Kraft or alkaline process. It produces a pulp of high physical strength and bulk, but relatively poor sheet formation. The process has a recovery system that not only recycles the pulping chemicals, but also is a source of about one-half of the steam generation. The yield of pulp is about 45 percent.

The largest single steam user in this type of paper mill is the black liquor evaporator that concentrates the black liquor sufficiently to burn it in the chemical recovery boilers. These evaporators represent superb heat pump opportunities and will provide numerous benefits to a mill that purchases boiler fuels (e.g., gas, oil, or coal.)

Sulfite Pulping. Sulfite pulping uses sulfurous acid and an alkali to produce pulps of lower physical strength and bulk, but exhibits better sheet formation properties. The yield on the basis of chipped wood is again about 45 percent. These pulps are blended with ground wood for newsprint and are used in printing, bond papers, and tissue. Sulfite pulping was originally designed with a recovery system similar to the older soda process still used in some plants. Environmental pressures have often forced these plants to develop a recovery process.

A revolutionary approach to chemical pulping seeks to replace the "conventional" Kraft process to eliminate both sulfur and chlorine. In this approach, wood chips would be chemically pulped using oxygen, or thermo-mechanically pulped and delignified by treatment with oxygen and alkaline solutions.

Mechanical and chemi-mechanical defibration methods.

In mechanical pulp production, chemical treatment is not used. Defibration is achieved using mechanical stressing and heating wood material. The energy brought to wood transforms to heat and enables separation of fibers by mechanical work. Combination of chemical and mechanical pulp production is called the chemi-mechanical method, CTMP, where chips are defibrated mechanically after a short chemical treatment.

Comparison of the kraft process with other pulping processes:

Pulp produced by the kraft process is stronger than that made by other pulping processes. Acidic sulfite processes degrade cellulose more than the kraft process, which leads to weaker fibers. Kraft pulping removes most of the lignin present originally in the wood whereas mechanical pulping processes leave most of the lignin in the fibers. The hydrophobic nature of lignin interferes with the formation of the hydrogen bonds between cellulose (and hemicellulose) in the fibers needed for the strength of paper (strength refers to tensile strength and resistance to tearing).

Kraft pulp is darker than other wood pulps, but it can be bleached to make very white pulp. Fully bleached kraft pulp is used to make high quality paper where strength, whiteness and resistance to yellowing are important.

The kraft process can use a wider range of fiber sources than most other pulping processes. All types of wood, including very resinous types like southern pine and non-wood species like bamboo and kenaf can be used in the kraft process.

Pulping (Cooking) processes can be divided into two main categories (in technological and apparatus arrangement). They are the *batch method* and the *continuous method*.

Historical notes. The use of wood to make pulp for paper began with the development of mechanical pulping in Germany by F.G. Keller in the 1840s. Chemical processes quickly followed, first with J. Roth's use of sulfurous acid to treat wood, followed by B. Tilghman's US patent on the use of calcium bisulfite, $\text{Ca}(\text{HSO}_3)_2$, to pulp wood in 1867. Almost a decade later the first commercial sulfite pulp mill was built in Sweden. It used magnesium as the counter ion and was based on work by Carl Daniel Ekman. By 1900 sulfite pulping had become the dominant means of producing wood pulp, surpassing mechanical pulping methods. The competing chemical pulping process, the sulfate or kraft process was developed by Carl F. Dahl in 1879 and the first kraft mill started (in Sweden) in 1890. The invention of the recovery boiler by G.H. Tomlinson in the early 1930s allowed kraft mills to recycle almost all of their pulping chemicals. This, along with the ability of the kraft process to accept a wider variety of types of wood and produce stronger fibers made the kraft process the dominant pulping process starting in the 1940s. Sulfite pulps now account for less than 10% of the total chemical pulp production and the number of sulfite mills continues to decrease.

I.1. Production of sulfate viscose pulp

The Kraft process describes a technology for conversion of wood into wood pulp consisting of almost pure cellulose fibers.

Purpose of pulping (cooking pulp) in chemical pulp production is to use chemicals and heat to remove fiber binding lignin so chips defibrate easily. Fibers containing cellulose are tried to keep as long, unbroken and strong as possible. Also wood extractives which can later cause foaming and precipitants in the process are tried to remove. Today sulfate cooking is the most commonly used pulp production method.

Chemicals which dissolve as much lignin and as little cellulose as possible are used in pulping. Sulfate process uses white liquor, a mixture of sodium hydroxide (NaOH) and sodium sulfide (Na_2S). Sodium hydroxide degrades lignin and sodium sulfide fastens cooking reactions and decreases cellulose degradation caused by sodium hydroxide. Temperature in sulfate pulping is normally 150 - 170 °C.

Lignin amount left in fibers is expressed with a kappa number. Lignin causes pulp to turn brown during cooking. Because bleaching chemicals are much more expensive than cooking chemicals, as much as possible of the lignin is tried to remove during the cooking process. However, too extensive lignin removal causes

cellulose degradation to increase. This decreases pulp strength and yield. Today, typical kappa number for pulp to be bleached is 14 - 20 for hardwood and 20 - 30 for softwood pulp. If the pulp is not beached, the kappa number after cooking will be much higher, typically 40 - 100. Pulp yield is typically 50 - 53% for hardwood and 46 - 49% for softwood.

Controllability and smoothness of the cooking process are requirements for succeeding of the following process phases. Digester plant faults reflect to other departments and cause changes for pulp properties such as strength, brightness and beatability changes, debris and brightness reversion.

Main parameters of the sulfate cooking processes are:

pulp quality

operating costs (energy, chemicals and wood consumption)

investment costs

environmental factors

process reliability

secondary product usefulness.

Wood chips are fed into vessels called digesters that are capable of withstanding high pressures. Some digesters operate in a batch manner and some in a continuous process, such as the Kamyr digester. Digesters producing 1,000 tonnes of pulp per day and more are common.

During batch process the pulp is cooked phase by phase in each digester. There are several digesters in a digester house. During continuous cooking chips and chemicals are continuously fed from the top and removed from the bottom of the digester. Digester is divided to zones, in which different phases take place.

Kappa number after cooking has decreased during last decades in order to minimize environmental load. For example in the 1970's the typical kappa number after cooking was 35 for softwood. Nowadays it's usually below 30. This decreases lignin amount to be removed during bleaching and consumption of bleaching chemicals. Because the removed lignin goes to bleaching line effluents, it is beneficial for reducing the effluent load to continue cooking as long as possible. However, there is no use to decrease the kappa number too much during the cooking, because the pulp strength properties and yield will decrease considerably after a certain point. Rapid development of oxygen delignification has caused that instead of trying to reach very low cooking kappa numbers, more lignin is removed during the oxygen phase.

Both batch and continuous cooking have several variations, which are usually based on various liquid changes during the cooking. The purpose of changing the reaction conditions is to improve pulp quality, to enable cooking to a lower kappa numbers and to decrease energy consumption.

The following four main rules are used when cooking further to lower kappa numbers without damaging the pulp properties:

Alkali concentration (NaOH and Na₂S) should be low in the beginning of cook and stay consistent during it. High alkali concentration in the beginning increases cellulose degradation and consumes alkali unnecessarily.

Hydrogen sulfide concentration (HS⁻, forms when Na₂S dissolves and reacts with wood) must be as high as possible in the beginning of the cook. Hydrogen sulfide increases lignin dissolving rate and it doesn't degrade cellulose in the beginning of cooking like sodium hydroxide does.

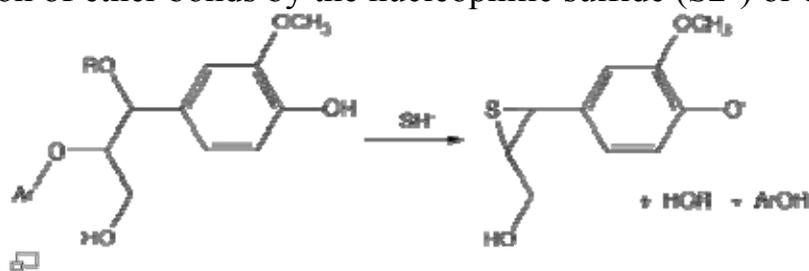
Dissolved lignin concentration should be as low as possible, especially in the end of cooking. Dissolved lignin slows delignification reactions. Moreover, it may re-precipitate on fiber surfaces, especially when alkali concentration reduces too much.

Temperature should be low, especially in the beginning of cooking. Low temperature decreases cellulose degradation. This degradation is especially strong in the beginning of cooking.

How cooking and fiber line processes treat fibers mechanically has also influence on pulp strength properties (for example pressure and heat shocks, mechanical strain and especially their combinations). These factors are under research at the moment.

The process is arranged as follows. Wood chips and white liquor, a mixture of sodium hydroxide and sodium sulfide, produced in the recovery process, are added to the top of the digester. In a continuous digester the materials are fed at a rate which allows the pulping reaction to be complete by the time the material exit the reactor. Typically delignification requires several hours at 130 to 180 °C (265 to 355 °F). Under these conditions lignin and some hemicellulose degrade to give fragments that are soluble in the strongly basic liquid. The solid pulp (about 50% by weight based on the dry wood chips) is collected and washed. At this point the pulp is quite brown and is known as "brown stock". The combined liquids, known as black liquor (so called because of its color), contain lignin fragments, carbohydrates from the breakdown of hemicellulose, sodium carbonate, sodium sulfate and other inorganic salts.

One of the main chemical reactions that underpin the kraft process is the scission of ether bonds by the nucleophilic sulfide (S²⁻) or bisulfide (HS⁻) ions.

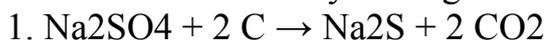


net reaction in depolymerization of lignin by SH⁻ (Ar = aryl, R = alkyl groups).

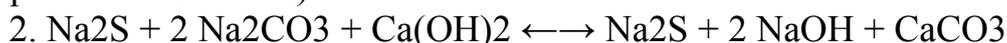
Recovery process:

The black liquor is concentrated in multiple effect evaporator to 60% or even 80% solids ("heavy black liquor") and burned in the recovery boiler to recover the inorganic chemicals for reuse in the pulping process. Higher solids in the concentrated black liquor increases the energy and chemical efficiency of the

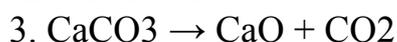
recovery cycle, but also gives higher viscosity and precipitation of solids (plugging and fouling of equipment). The combustion is carried out such that sodium sulfate is reduced to sodium sulfide by the organic carbon in the mixture:



The molten salts ("smelt") from the recovery boiler are dissolved in a process water known as weak wash. This process water also known as weak white liquor is composed of all liquors used to wash lime mud and green liquor precipitates and is kept in a tank called weak wash storage tank. The solution of sodium carbonate and sodium sulfide resulted is known as "green liquor". This liquid is mixed with calcium hydroxide to regenerate the white liquor used in the pulping process through an equilibrium reaction (Na₂S is shown since it is part of the green liquor, but does not participate in the reaction):



Calcium carbonate precipitates from the white liquor and is recovered and heated in a lime kiln where it is converted to calcium oxide (lime).



Calcium oxide (lime) is reacted with water to regenerate the calcium hydroxide used in Reaction 2:



The combination of reactions 1 through 4 forms a closed cycle with respect to sodium, sulfur and calcium and is the main concept of the called recausticizing process where sodium carbonate is reacted to regenerate sodium hydroxide.

The recovery boiler also generates high pressure steam which is led to turbogenerators, reducing the steam pressure for the mill use and generating electricity. A modern kraft pulp mill is more than self-sufficient in its electrical generation and normally will provide a net flow of energy to the local electrical grid. Additionally, bark and wood residues are often burned in a separate power boiler to generate steam.

Key technical characteristic of **Kraft process** are as follows:

Full chemical pulping with NaOH and Na₂S, pH above 12, 160-180°C, 800 kPa (120 psi), 0.5-3 hrs. Useful for any species of wood, high strength of pulp, tolerant to bark, efficient energy and chemicals recovery cycle
Disadvantages: difficult to bleach compare to sulfite pulps, low yields (carbohydrate losses), sulfur compounds- extremely offensive odor 80% of lignin dissolved, 50% hemi, 10 % of cellulose

Grades:

Soft cooks- (for bleached grades) lignin content 3.0-5.2 % (20-35 Kappa number)

For softwoods, or 1.8-2.4 % lignin (12-18 Kappa number) hardwoods.

Medium soft cooks- (bag papers) lignin- 5.2-7.5% (35-50 Kappa number).

Hard softwood cooks- 9-11 % lignin (65-70 Kappa number)- top liner boards 12-16.5 % lignin (80-110 Kappa number) – bottom liner.

Digester: Pressurized vessel for cooking chips into pulp.

A. Batch Digester

70-350m³

Mill has a bank of 6-8 digesters

Heating: Direct- with the steam, dilutes the cooking liquor, changes the liquor to wood ratio, bad uniformity of cooking-disadvantage Indirect- cooking liquor heated outside the digester, heat exchanger. More uniform heating and cooking.

Cooking time: time from initial steaming of chips to start of digester blowing

Time to temperature: Time from initial steaming of chips to the point when desired temperature is reached

Time at temperature: Time from when the cooking T is reached until digester blow starts

Cooking sequence:

- Filling of the digester with chips, white liquor, and weak black liquor
- After initial circulation of the liquor (contents settle) additional chips are added
- Digester sealed and heating starts; temperature rises for about 90 min until the cooking temperature is achieved.
- Cooking T is reached- cook at the temperature 0.5-3 hrs
- During the heating time, air and other gases from the digester are vented
- When cook is completed (Kappa number), content of the digester is discharged to the blow tank
- Digester is opened and the sequence is repeated

B. Continuous Digester

Tube shaped vessel, with elements: presteaming, liquor impregnation, heating, cooking, and washing Chips enter and exit the digester continuously More space efficient, easier to control, labor saving, energy efficient than batch digester Rotary valves help to fill the digester, from atmospheric to high pressure

KAMYR DIGESTER

Vertical digester (one to two vessels) 77 million tons/year
3 zones: heating, cooking, washing

M&D Digester (Messig & Durkee)

Inclined at 45 degree
Kraft pulping of sawdust, acid prehydrolysis (with kraft), and semichemical pulping
Cooking time approx. 30 min
Diameter about 2.4 m- relatively small production levels

PANDIA DIGESTER

Horizontal digester, multi- tube (2-8 digester)

Screw feed

Kraft pulping of sawdust

Semichemical pulping of chips, non-woods (straw), short cooking times

*Blow tank**Digester blowing*

Continuous digester- @ about 100°C

Batch digester- @ or near cooking temperature 170 °C, pulp loses a significant amount of strength (10-15%)

Vapor expansion in chips is usually sufficient force to cause fiber separation

Liquor to wood ratio

3:1 to 4:1 in full chemical pulping

It is kept as small as possible while maintaining good digester operation including good circulation for even cooking

$\text{LIQUOR/WOOD} = \frac{[\text{TOTAL PULPING LIQUOR MASS}]}{[\text{DRY WOOD MASS}]}$

Terminology in the sulfate pulping.

Cooking chemicals:

White liquor is a chemical mixture used in sulfate pulping. The effective chemicals of it are sodium hydroxide (NaOH) and sodium sulfide (Na₂S). The concentration of those compounds in white liquor is expressed as affecting e.g. active alkali or effective alkali (g/l).

Black liquor is white liquor which has reacted in digester and to which wood compounds have dissolved. Black color comes from lignin compounds colored by alkali and dissolved to liquor.

Sodium hydroxide and sulfide are expressed in grams per liter of sodium hydroxide or sodium oxide (Na₂O) equivalents. Practice is based on sodium contents of the compounds. Conversion factor from Na₂O to NaOH is 1.29 and 0.775 in reverse direction.

Green liquor is recovery boiler smelt dissolved to weak white liquor. In other words it is black liquor with organic incinerated. In addition other reactions have taken place, for example sodium sulfate has transformed to sodium sulfide. Green liquor is processed to white liquor in recaustizing plant.

Sodium sulfide concentration in cooking liquor is expressed as sulfidity (%). Sulfidity is usually on the level 35 - 45% in modern mills. Reduction (%) shows how completely the nearly inert sodium sulfate has been reduced to useful sodium sulfide. Reduction takes place in recovery boiler. Causticity (%) shows chemical efficiency of white liquor production (causticization). It shows how much inert sodium carbonate has been transformed to useful sodium hydroxide.

Effective alkali, active alkali and sulfidity are the most important properties of white liquor. Effective alkali indicates OH⁻ ion concentration, active alkali total amount of OH⁻ and HS⁻ ions and sulfidity HS⁻ and OH⁻ ion ratio.

Alkali charge is usually expressed as percentages of wood. This means alkali amount in relation with completely dry wood.

Due to reaction balance during white liquor production the white liquor concentration is approximately 140 - 170 g/l active alkali as NaOH. White liquor contains also other sodium salts, such as sodium sulfate (Na₂SO₄) and sodium carbonate (Na₂CO₃) and small amounts of sulfites and chlorides. All sodium salts can be expressed as total alkali (TTA, titrating alkali, g/l). All sodium compounds are taken into account, such as sodium sulfate and carbonate. Large amounts of sulfate and carbonate in white liquor indicate malfunctions in recovery boiler or recausticizing plant. Because sulfate and carbonate don't significantly participate in cooking processes, they are only unnecessary loads in chemical circulation. The white liquor contains also other substances not reacting in cooking, such as chlorides and calcium compounds. The amount of these so called inert materials depends greatly on mill chemical circulation, for example on white liquor filtration success.

Anthraquinone (AQ) is an organic compound, which has been found to increase cooking yield, especially for lower sulfidity levels. In high sulfidity mills, such as running at 40%, the difference is much lower. High cost of anthraquinone has for now decreased its' use. However, in some cases when a mill's chemical circulation is a bottleneck, its' use has been found to be profitable.

Cooking control and reactions.

H-factor indicates relative speed of lignin dissolution. It depends on cooking time and temperature. H-factor's dependency on temperature is very strong due to delignification temperature dependency. Even a difference of couple of degrees in cooking temperature can make a big difference in pulp quality. H-factor has been defined so that 1 hour in 100 °C is equivalent with H-factor 1.

Liquor-to-wood ratio indicates the total liquid amount compared to completely dry wood. It includes all liquids involved in cooking; cooking liquor, possible supplementary liquor and water contained in chips after possible presteaming.

Presteamming means treating chips with steam before cooking. Steam treatment removes air from chips pores and helps cooking chemicals absorption in the beginning of cooking.

Flashing means that hot (130 - 170°C) black liquor from a continuous digester is released from digester to lower pressure. This causes the liquor to boil, which forms steam containing odorous gases.

Carbohydrates are sugar compounds, which form cellulose and hemicelluloses. Delignification means lignin dissolving in cooking liquor. Condensation means lignin reprecipitation from cooking liquid onto fiber surface. Residual alkali indicates amount alkali, which is in cooking liquor after cooking.

VOC compounds (Volatile Organic Compounds) are uncondensing hydrocarbons, which form in chips presteaming, cooking reactions and in all black liquor treatment processes.

TRS compounds (Total Reduced Sulfur) are uncondensing reduced sulfur compounds. In the other words they are VOC compounds containing sulfur. TRS

compounds form in cooking reactions and in every black liquor treatment phase. TRS compounds have a strong smell. Even in low concentrations they cause the smell typical for pulp mills. However, odor problems have decreased considerably with modern technology (odorous gas collecting and burning).

Notice that, to obtain pulp for chemical processing, having rather high reactivity, alkaline sulfate cooking with preliminary chip hydrolysis (two-stage combined process) is commonly applied. Two types of the pre-hydrolysis are applied: acid, using weak sulfuric or hydrochloric acids (to pH 1-1.5) at 100-125°C for 2-5 hours, or water, which finds much wider commercial application (at pH 3-4, 140-180°C for 0.3-3 hours). Sulfate cooking with water pre-hydrolysis with following multi-stage bleaching is used in many countries to obtain pulp for following production of fiber, including viscose. Instead of the water pre-hydrolysis, deep presteaming of chip can be used.

This process version is applied at **Baikalsk PPM** (Baikalsk Pulp&Paper Mill, BPPM, Irkutsk region) in production of *sulfate viscose pulp*.

One of the most important parameters of viscose pulp is content of alfa-cellulose. Sulfate cooking with water pre-hydrolysis allows to obtain unbleached refined pulp, containing 95-96% of alfa-cellulose.