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(54) **Retting procedure for bast plants such as: flax, hemp, jute, etc.**

(57) The procedure, according to the invention, consists of the fact that the retting of bast plant stems is done in vats by using a mixed inoculum of microorganisms from a substrate of seedy fruit and citric waste and baker's yeast (*S. cerevisiae*), with added $\text{AlK}(\text{SO}_4)_2$ in a buffer medium with a pH of 4,5 achieved using citric acid/potassium citrate, for a period of 48 hours at a temperature of 30°C, and a hydromodule of HM 1:17. Waste water treatment is done through adding a symbiotic culture composed of yeasts and acetic bacteria in a concentra-

tion of 1:1000 compared to the fleet. The treatment phase consists of maintaining this culture in the waste water for 3-4 days at ambient temperature, after which the culture is removed and the water is reinserted into the technological process or if discharged. Using this procedure, an extraction yield of +/- 30% superior quality fibers is ensured, as well as the possibility of reusing waster in the process.

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Description**Field of the invention**

[0001] The present invention refers to a procedure for retting bast plant stems such as flax, hemp, jute etc. If flax or hemp fibers are to be used for textiles or other top range applications, then these must be separated from the rest of the stem. Retting is a microbial process through which the chemical bonds which connect the fibers and the ligneous part of the stem are broken. Retting may be achieved through various means: using dew (the harvested stems are left out in the field to rot until the fibers separate from the ligneous part, a process which can last for weeks) or using water (stems are immersed in water basins or in rivers for 7-15 days) and monitored until the fibers are separated. The latter process requires large amounts of water which must be treated afterwards, though it yields fibers of highly superior quality compared to those retted using dew.

[0002] The problem posed by bast plant retting is, on the one hand, the processing time, and on the other hand, the quantity and quality of the fibers obtained after the separation process (the fiber/stem ratio). Furthermore, the quantity and quality of the wastewater which must be treated before being released out into the environment must be considered, due to its high concentration of organic compounds.

Background of invention

[0003] Over time, specialists have speculated whether water retting may offer more efficient and/or more ecological solutions, by using additional microorganisms, specially selected, or even by directly using enzymes. A few examples of patented retting methods are given below:

a) WO/2011/049258 - Bast plant retting method which uses *ENTEROBACTER GERGOVIAE* HL 2006 which is one of the few microorganisms found on the surface of hemp which has a superior effect in destroying pectin and lignin compounds. Patent: 28.04.2011, PCT/KR2009/006335SKYNEUPHARM. CO., LTD.PARK, Sun Kyu

b) KR1019910004452 - flax retting method using clusters of gram-negative microbes. Patent, 29.06.1991. The method implies retting flax or hemp in a treatment vat where air is bubbled at a temperature of 23-27 ° C, pH 8- 8.5 for 60-80 hours. Under these conditions, the gram-negative microbe complex (*Erwinia tracheiphila*, *Erwinia Salicis*, *Erwinia stewartii*, and *Enterobacter agglomerans*) is activated by the oxygen and favors the breakdown of pectin compound.

c) WO WO/1987/002390 - Patent 23.04.1987, PCT/FR1986/000358COMITE ECONOMIQUE AGRICOLE DE LA PRODUCTION DU CHAACKAWI, Jean-Sony: The method implies using SPS-ase enzymes.

[0004] Excepting the patented methods, specialized literature abounds with studies regarding other retting methods, where various bacteria were used in order to reduce melting times and to improve fiber quality. Di Candilo M and others, in their work, entitled "Effects of selected pectinolytic bacterial strains on water-retting of hemp and fiber properties", J Appl Microbiol. 2010 Jan; 108(1):194-203., show that, using two pectinolytic bacteria (*Clostridium* sp. L1/6 and *Bacillus* sp. ROO40B) in the retting process of hemp stems accelerates the process and helps improve fiber quality significantly. The best results are seen after 3-4 days of treatment.

[0005] Another study led by QIAN Wei-jun and collaborators, in "Influence of the water quality on the hemp degumming effect", JOURNAL OF TEXTILE RESEARCH » 2006, Vol. 27 » Issue (6): 19-22, refers to the possibility of replacing freshwater with saltwater for retting" through their quality is inferior to those which undergo the classic solution.

The solution to the technical problem

[0006] The present invention proposes a complete solution for the retting process of bast plant stems in vats, by using an ecological treatment formula with mixed inoculum of microorganisms from a substrate of fruit waste and baking yeast, including biological treatment of wastewater based on the use of symbiotic cultures composed of yeasts and acetic bacteria. The retting process is done in vats, at a hydromodule of 1:17, temperature 30°C, in the presence of a mixed inoculum of microorganisms from a substrate of seedy fruit or citric waste and baker's yeast (*S. cerevisiae*), with added $\text{AlK}(\text{SO}_4)_2$ in a pH 4,5 buffer medium composed of citric acid /sodium citrate, for a period of 48 hours. The biological treatment process of waste water consists of adding the symbiotic culture mentioned above at a rate of 1:1000 (in relation to the fleet) to the waste water and maintaining it in these conditions for 3-4 days at ambient temperature, after which the culture is removed and the water is returned to the technological process or is discharged. Through this procedure, an extraction yield of +/- 30% fibers of superior quality is obtained, as well as the possibility of reusing the waste water in the process.

[0007] The technical problem that the invention solves consists of:

a) Using a mixture of self-adapting microorganisms in the retting phase, which are capable of producing an efficient enzymatic cocktail for decomposing pectins which are present in the substrate (stems). This inoculum, in the presence of auxiliaries, is able to accelerate the retting process at a temperature of 30°C so that the separation of the fibers from the stems take place in 48 hours, the extraction yield fibers/stems be 30% and the fibers fit 1st class quality standards. The auxiliaries used (citric acid, sodium citrate and potassium alum) during the retting process and in the concentrations given do not represent environmental pollutants.

b) The combination of inoculum/auxiliaries in the working conditions given does not allow for putrefactive bacteria, which would otherwise lead to foul smelling emanations, like in the classic procedure;

c) Use of a symbiotic culture during the treatment phase, which assimilates the excessive pectins present in the waste water, allowing for its reuse or discharge.

Detailed description of the invention

[0008] The retting procedure for bast plants, according to the invention, removes the disadvantages of existing similar procedures (those that used selected microorganisms) through the fact that, by using this special mixed inoculum, the isolation, selection and purification stages for the specially formulated microorganisms are removed. The process takes place at a temperature of 30°C and a pH 4.5, which is not claimed by any other patented or published procedure. This pH and the presence of alum in the reaction medium ensure the controlled selection of the microbial species capable of accelerating the process and inhibit the development of unwanted species. The elimination of competing species leads to the finalization of the retting process in a short amount of time and avoids the development of secondary compounds which affect fiber quality (color, smell, flexibility). The low pH also inhibits the development of putrefactive bacteria (whose optimal pH lies in the neutral/slightly alkaline range) which generate metabolic products with the characteristic smell of the classic procedure.

[0009] Due to the fact that the inoculum used is obtained through the use of seedy fruit and citric waste, combined with baker's yeast, the method is an ecological, economical, efficient and sustainable solution.

[0010] Additionally, during the treatment phase of the waste water resulting from the first stage (whose pH has ultimately reached a pH of 6,5 - 7,0 after 48 hours) a symbiotic culture is used, one which requires no added chemical reagents, which would prove costly and aggressive to the environment. The treatment is carried out in the conditions existing *in situ*, without need for any of the operations found in classic treatments (decantation, precipitation, aeration). The symbiotic culture does not require any additional investments for maintenance and regeneration, given its ability to increase its biomass by consuming the pectins present in the waste water. At the end of the treatment process the symbiotic culture is extracted from the vat for further use.

Presentation of advantages

[0011] The invention presents the following advantages:

- Using this special mixed inoculum eliminates the isolation, purification and selection phases for the specially formulated microorganisms;
- The process is not energy intensive, as it takes place at a temperature of 30°C
- The pH of 4.5, and the presence of alum in the reaction medium ensure the controlled selection of the useful species and the inhibition of unwanted species;
- At the end of the retting process, the reaction medium's pH is 6,5-7,0;
- The retting process is accomplished quickly (48 hours);
- No putrefactive bacteria develop, which would produce the foul smelling emanations typical of the classic process;
- The method implies superior revaluation of seedy fruit and citric waste;
- Treatment is performed using a symbiotic culture;
- During treatment, no costly or environmentally aggressive chemical reagents need to be added;
- The possibility of reusing the treated waters in the procedure;
- Treatment is economical, due to the fact that no technological operations such as decanting, precipitating, aerating etc. are needed, and no costs are registered for maintaining the symbiotic culture;

Example

[0012] An example of invention application follows.

[0013] Consider a quantity of approximately 30 kg of hemp stems which are immersed into a vat which contains 500 liters of buffer solution composed of citric acid/sodium citrate, pH = 4.5, at a temperature of 30°C, to which approximately 1.2 kg of fruit waste and baker's yeast (*S. cerevisiae*) and 150 g potassium alum are added. The stems are kept in these

reactive conditions for 48 hours, after which they are extracted, washed and dried. The resulting waste water is transferred to a treatment vat where 500 g of symbiotic yeast and acetic bacteria cultures are added. After 3-4 days, the symbiotic culture is removed and the water is reinserted into the technological process or is discharged. After mechanical separation of the fibers from the stems, a yield of approximately 30% is obtained. The quality of the fibers separated from the hemp stems thus treated is evaluated through the strand breaking load, length density and flexibility. The results obtained are presented in the following table:

Physical-mechanical characteristic	Value	Method of analysis and trial	Notes
Strand breaking load (N)	330	STAS 6183/2-69	Superior quality
Length density Nm (tex)	74 (13,5)	STAS 8468-69	Superior quality
Flexibility (mm)	41	-	Superior quality

Claims

1. The procedure of retting of bast plants, such as flax, hemp, jute, etc., according to the invention, it is being **characterized by** the fact that it is accomplished in two phases, in the first phase takes place the retting of the bundles of stalks by immersing them for 48 hours into a solution that contains: buffer consisting of citric acid / sodium citrate, pH = 4,5, 1-2 kg fruits waste, 100-200 g bakery yeast, 150-300 g potassium aluminium sulphate with a liquid ratio straw to solution of 1:17 and a temperature of 30-35°C, followed by rinsing and drying, and in the second phase takes place the cleaning of the waste water by adding symbiotic cultures of yeasts and acetic bacteria in a ratio of 1:1000 towards the fleet, with a duration of 3-4 days at the room's temperature until the pectin are reduced, followed by the collecting of the symbiotic culture of yeasts and the reintroduction of the cleaned water in the retting process.
2. The procedure according to claim 1, **characterized by** the fact that the retting is made by the mixture consisting of fruits waste, bakery yeast and potassium aluminium sulphate within 48 hours.
3. The procedure according to claim 1, **characterized by** the fact that the removal of pectin from the waste water is accomplished with the help of the symbiotic culture of yeasts and acetic bacteria without adding other chemicals and other specific operations characteristic to classical waste water treatments (decantation, precipitation, aeration, etc.).
4. The procedure according to claim 1, **characterized by** the fact that the waste water are reintroduced into the process.



EUROPEAN SEARCH REPORT

Application Number
EP 13 46 4005

DOCUMENTS CONSIDERED TO BE RELEVANT			
Category	Citation of document with indication, where appropriate, of relevant passages	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
A	CN 101 624 727 A (UNIV HUAZHONG SCIENCE TECH) 13 January 2010 (2010-01-13) * claim 1 *	1-4	INV. D01C1/04
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A	M. DI CANDILO ET AL: "Effects of selected pectinolytic bacterial strains on water-retting of hemp and fibre properties", JOURNAL OF APPLIED MICROBIOLOGY, vol. 108, no. 1, 26 May 2009 (2009-05-26), pages 194-203, XP055091610, ISSN: 1364-5072, DOI: 10.1111/j.1365-2672.2009.04409.x * the whole document *	1-4	
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The present search report has been drawn up for all claims			TECHNICAL FIELDS SEARCHED (IPC) D01C
Place of search The Hague		Date of completion of the search 9 December 2013	Examiner Verschuren, Jo
CATEGORY OF CITED DOCUMENTS X : particularly relevant if taken alone Y : particularly relevant if combined with another document of the same category A : technological background O : non-written disclosure P : intermediate document T : theory or principle underlying the invention E : earlier patent document, but published on, or after the filing date D : document cited in the application L : document cited for other reasons & : member of the same patent family, corresponding document			

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**ANNEX TO THE EUROPEAN SEARCH REPORT
ON EUROPEAN PATENT APPLICATION NO.**

EP 13 46 4005

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report.
The members are as contained in the European Patent Office EDP file on
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For more details about this annex : see Official Journal of the European Patent Office, No. 12/82

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