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Toilet paper recovery from municipal wastewater and application in building sector

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Abstract: One of the most innovative applications for a circular economy approach is the recovery of cellulose fibres from municipal wastewater. Recovered cellulose fibres from the wastewater could bring benefits to the construction industry in terms of reducing the amount of non-renewable raw materials and increasing sustainability. Rotating belt filter was used to obtain cellulose fibre-rich sludge from real urban influent. Recovered cellulosic material reached values up to 26.59 g m^{-3} when the solids removal efficiency was higher than 70%. Treated cellulosic sludge had an average of 87% content of cellulose, hemicellulose and lignin. The recovered cellulose fibres were analysed and used in mortar mix to understand their possible impact in the building sector and the effect on the properties of hydraulic lime-based mortars. Properties of fibre addition were investigated in terms of microstructure and mechanical strength. Cellulose fibres were added by mix volume up to 20%. The overall results with the maximum content of cellulose fibres indicated the improvement of mortars performance in terms of increased lightness, flexural strength and hygrometric properties.

Keywords: Cellulose recovery, municipal wastewater, mortars, sustainable construction

Table 1. List of acronyms used in the paper.

Acronyms	Meaning
COD	Chemical Oxygen Demand
CREC	Mortar with recovered fibres addition
E%TSS	Efficiencies of the separation based on total suspended solids removal
EDAX	Energy Dispersive X-ray
$\mathbf{H_{max}}$	Maximum Hydraulic Level
$\mathbf{H_{min}}$	Minimum Hydraulic Level
HLR	Hydraulic Loading Rate
ITZ	Interfacial Transition Zone
MBC	Moisture Buffering Capacity
MBV	Moisture Buffering Value
Q	Flow rate
RBF	Rotating Belt Filtration
$\mathbf{R_c}$	Compressive Strength
$\mathbf{R_f}$	Flexural Strength
REFERENCE	Mortar without cellulose fibres addition
RH	Relative Humidity
sCOD	Soluble Chemical Oxygen Demand
SEM	Scanning Electron Microscope
SLR	Solids Loading Rate
SSD	Saturated Surface Dry
T	Temperature
TS	Total Solids
TVS	Total Volatile Solids
TSS	Total Suspended Solids
WVP	Water Vapour Permeability
WWTP	Wastewater Treatment Plant
$\mathbf{Y_{cm}}$	Cellulosic material recovery yield
$\boldsymbol{\rho}$	Density
$\boldsymbol{\mu}$	Water vapour diffusion resistance factor

1. Introduction

Recently, many technologies, especially in the wastewater sector, have evolved to fulfil the goals that the circular economy proposes. The need to close the loops of the production market highlighted the necessity to apply the *"best available technologies"* to recover resources also from "waste" flows [1][2][3].

A possibility of achieving circularity targets and increasing sustainability of productive processes is given by the recovery of cellulosic material derived from toilet paper into wastewater. The benefits that could be derived from its recovery may be translated not only into the possibility of re-use it as a secondary raw material, but also as reinforcing component in binder-based materials [4][5][6] in engineering applications and especially in the building sector.

In this field benefits, in terms of mechanical characteristics and ability to improve indoor comfort, are well known for using cellulose fibres from other industrial activities [7], but nowadays, from the author's knowledge, the potential of using cellulosic material recovered from real wastewater treatment plant has never been explored.

For this reason, the scope of this paper is the study of cellulosic material recovery from real urban wastewater and its exploitation in the building sector and of the effect of fibres, added at different percentages, on the properties of hydraulic lime-based mortars.

2. Materials and Methods

2.1. Cellulose recovery in wastewater treatment plant
The study involved the recovery of cellulosic material in two real wastewater treatment plants (WWTPs) in Falconara Marittima and in Carbonera (Italy).

In the WWTPs, a Rotating Belt Filtration unit RBF (Salsnes SF 1000 by Trojan Technologies) was used and fed by the effluent from the pre-treatment of the full-scale plant (flowrate from 15 to 50 m^3h^{-1}). In the WWTPs 35 experimental tests of cellulosic sludge separation were conducted at different operative parameters for the investigation of their influence on the solid separation efficiencies. In particular, minimum and maximum hydraulic levels (H_{min} and H_{max}) in the filtration chamber were varied from 75 to 260 mm; different belt porosities from 350 to 90 µm were used.

For each test, the Total Suspended Solids (TSS) removal efficiency was related to the Hydraulic Loading Rate (HLR) expressed as m^3 h^{-1} for m^2 of filtration area and to the Solids Loading Rate (SLR) defined as kg TSS h^{-1} for m^2 of filtration area.

The characterization of all the matrices was carried out according to standard methods for the Examination of Water and Wastewater [8].

2.2. Cellulosic Sludge Recovery
The amount of cellulosic material (cellulose, hemicellulose and lignin) was determined by analysing sludges separated from the RBF. Cellulosic sludge samples were collected from different tests conducted with 350, 250, 150 and 90 µm meshes.

As first step of analysis, sludge samples were collected at WWTPs and they were prepared in laboratory by sieving and washing them with tap water for 30 min at 63 µm in order to obtain sludge more concentrated in cellulose fibres and devoid of the finest aggregates. Hereafter samples have been dried at 105 °C to obtain the dry matter, expressed as percentage of Total Solids (TS) to the weight of the whole sample, and at 550 °C for the volatile content, expressed as percentage of Total Volatile Solids (TVS) to TS ratio, according to Standard Methods (TVS/TS %).

With this procedure it was possible to obtain the weight of the TS (in grams) in the sludge, separated from the WWTP pre-washing (expressed as g TS) and the amount of the TVS in the sludge, separated from the WWTP post-washing (expressed as g TVS).

The ratio between TVS (post-washing) and TS (pre-washing) was used to determine the Cellulosic Material Recovery Yield (Y_{cm}).

For the second step of analysis separated sludges from pilot plant (dried at 105 °C), were used to determine the percentages of proteins, lipids, ashes, hemicellulose, pure cellulose, lignin and humic compounds according to a well optimised analytical method [9]. Only %hemicellulose, %cellulose and %lignin were considered for Y_{cm}. Result from both stages were linked to determine Y_{cm}.

2.3. Recovered cellulose in the building sector application
For the impact assessment in the building sector, recovered cellulosic fibres from wastewater were added at different percentages to several mortars to test and analyse the fibre influence on properties of mortar.

CirTec BV, a partner of SMART-Plant Project, have provided the cellulose fibres that have been used to manufacture all the mortars, to satisfy the amount of cellulose required. Fibres and subsequently mortars have been morphologically investigated by a Scanning Electron Microscope (SEM-EDAX).

To define mix-designs, the estimation of the amount of water absorbed by cellulose fibres to reach the Saturated Surface Dry (SSD) condition was calculated, according to well tested analytical method [10].

Mortars were prepared adding fibres content in the mix from 0%, 5%, 10%, 15% up to 20% by mortar volume [11][12] [13] .

To point out the effect of different fibres addition on the mortars, analysis were carried out on both, fresh mortars to determine the workability (considering the slump value) [14], and on 28-days hardened mortars to investigate the compressive and flexural strength (R_c and R_f) [13]. Mortar specimens' density ρ (g dm^{-3}) was calculated [13] and the porosity have been analysed with Mercury Intrusion Porosimetry (MIP) (Thermo Fisher, Pascal series 240).

Since many studies have been conducted in the past [15][16][17][18] on natural cellulose fibres (i.e. cellulose derived from hemp) capacity to enhance the water vapour permeability and the humidity storage, it was considered interesting to evaluate the same potential for lime-based mortars with addition of recovered cellulose from WWTP.

Thanks to properties mentioned above, the indoor microclimate could enhance having positive effect on human health [19][20] and the use of fiber in mortars could reduce the building energy expenditure [21].

For this purpose, prepared mortars were analysed in terms of Water Vapour Permeability WVP (in static conditions), [22] [23] and Moisture Buffering Capacity MBC (in dynamic conditions), assessed by a simplified version of the NORDTEST method [24].

The average WVP results are reported in terms of the water vapour diffusion resistance factor µ, defined as the ratio between the vapour permeability of stagnant air (kg $(Pa\ m\ s)^{-1}$) and the vapour permeability of the material (kg $(Pa\ m\ s)^{-1}$) at the same temperature and pressure [25]. The moisture buffering value (MBV expressed in g $(m^2\%\ RH)^{-1}$) was calculated as the amount of moisture changed by the material per surface unit and Relative Humidity (RH) gradient [26].

3. Results and Discussions

3.1. Separation yields of Rotating Belt Filtration

Efficiencies of the separation based on TSS removal (E%TSS) obtained with the rotating belt filtration units in the WWTPs ranged from 11% to 75%. In particular, it was found that the higher the SLR, the higher the efficiencies, as shown in Table 2.

Table 2. E%TSS trend with the variation of SLR.

SLR (kg TSS $m^{-2}h^{-1}$)	E%TSS (%)
0 - 10	11
10 -20	35±13
20 - 30	31±22
30 - 40	55±13
40 - 50	49±22
50 - 60	59±23
90 - 100	75
100 - 110	71

Possible explanation of the lowest yields could be attributed to the influent flow characteristics of the two WWTPs as shown in Table 3. Falconara influent values of the TSS (112±82mg l^{-1}) and the Chemical Oxygen Demand (COD of 192±140 mg l^{-1}) were lower than Carbonera values, which were TSS = 219±119mg l^{-1} and COD = 444±191 mg l^{-1}, respectively. Further, Falconara influent had a higher conductivity value (1432±96 µS/cm) than Carbonera (1064 µS/cm), probably due to the intrusion of seawater mainly during the rainy season, because Falconara WWTP is located near the coast. This condition could negatively have affected the separation yield with a dilution of the incoming wastewater to the RBF.

Table 3. TSS Removal Efficiencies for Falconara and Carbonera WWTPs.

pH	Alkalinity	Conductivity	TSS	VSS	VSS/TSS	COD	sCOD	
-	mg/l	µS/cm	mg/l	mg/l	%	mg/l	mg/l	
7.3±0.1	359±33	1432±96	112±82	-	-	192±140	38±11	Falconara
7.6±0.4	312±23	1064	219±119	194±118	91±7	444±191	-	Carbonera

Further, separation results from different tests revealed that different parameters could influence the separation efficiencies.

On the one hand, enhancements of E%TSS were reachable with a lower mesh of the RBF unit, as confirmed by efficiencies of 37±19% and 74±2% for 350 and 90 µm mesh, respectively.

On the other hand, solids separation was affected even by the specific SLR (kg TSS m^{-2} h^{-1}). Experimental results highlighted that, at the same belt mesh, for SLR in the range of 25 - 40 kg TSS $m^{-2}h^{-1}$, best TSS removal (E%TSS) were obtained, whereas for values lower than 20 kg TSS $m^{-2}h^{-1}$ and for range 40-108 kg TSS $m^{-2}h^{-1}$ E%TSS was not optimised.

E%TSS were correlated even with the solids concentration of the influent (TSS_{IN}) and the tested flow rates (Q). The result was that TSS removal was more sensitive to the influent TSS concentration rather than to flow rates because of the automatic adjustment of the belt speed [27].

Even the hydraulic level into the filtration chamber affected the E%TSS. In fact, for results of tests with hydraulic levels H_{max} = 260 mm and H_{min} = 240 mm, the E%TSS was averagely 48±22%, while for tests with H_{max} = 220 mm and H_{min} = 200 mm the E%TSS was on average 57±17%. Therefore, by decreasing the hydraulic capacity, TSS removal performance was enhanced.

All these results highlighted that possible optimization of TSS removal, for cellulose fibre-rich sludge, is achievable by controlling RBF operative parameters in order to positively affect the separation of solids and fibrous material during the filtration process.

3.2. Cellulosic material recovery yields

From the separation tests by RBF, sludge samples were collected in order to analyse their cellulosic material content. It has been shown that sludges had different amounts of ashes, lipids, cellulose, hemicellulose, lignin and humic compounds, protein and other compounds.

Further, the washing process of cellulosic sludge itself influenced the composition because during the washing part of lipids, protein and fine aggregates were removed. The effects are shown in Table 4.

Table 4. Average percentages of main cellulosic sludge components before and after washing.

Component	Non washed sludge (%)	Washed sludge (%)
Ashes	15±13	4±1
Lipids	10±4	9±1
Cellulose	28±11	38±6
Hemicellulose	7±1	9±1
Lignin and Humic compounds	26±3	40±3
Protein	12±12	-
Other compounds	2	-

The value of g TVS (post-washing) to g TS (pre-washing) ratio was resulted averagely equal to 0.8±0.1. This value and the percentages of cellulose, hemicellulose and lignin, were used to determine the cellulosic material recovery yield (Y_{cm}).

It has been found that the higher E%TSS, the higher Y_{cm}. In particular, average values of Y_{cm} varied in E%TSS different ranges as shown in Table 5. Further, the average Y_{cm} reached values up to 26.59 g $_{\text{cellulosic material}}$ m^{-3} (averagely 7.8 ± 6.9 g $_{\text{cellulosic material}}$ m^{-3}).

Table 5. Results of Y_{cm} related to E%TSS.

E%TSS range (%)	Y_{cm} (g $_{\text{cellulosic material}}$ m^{-3})
0 - 20	0.4
20 - 40	5.3±3.2
40 - 60	7.2
60 - 80	12.6±8.8

3.3. Fibres characterization and effect of recovered cellulose on mortars
Recovered fibres are shown in Figure 1. Their characteristics and the morphological aspect were analysed by SEM. The surface of fibres appeared rough and the aspect is tube-shaped (Figure 2).

Figure 1. CREC fibers.

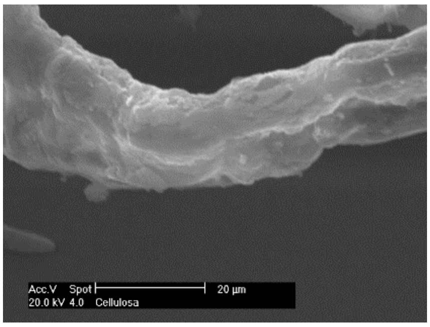

Figure 2. Surface of CREC fibres.

Further, fibre diameter and grain sizes were analysed. Distribution diameter curve showed the diameter appears thin and with an average value equal to 11.1 µm.

In the examined fibres, the EDAX analysis, not considering the main elements as C, H, O, gave different relative percentages by weight of metal elements (Al, Si, S, K, Ca and Fe), as reported in Table 6.

Table 6. EDAX analysis of cellulose fibres.

Elem	CREC (wt%)
Al	0.8
Si	2.0
S	0.1
K	1.0
Ca	94.1
Fe	1.8
TOTAL	100

The water absorption of recovered fibres was 119% in order to achieve the SSD condition. The measured apparent density of recovered cellulose was equal to 0.46 g cm^{-3}. Different fibre amounts were added to the blends to test different mortar mixes.

The main effect caused by fibre addition was the reduction of mortar slump values [29] which ranged from 118(REFERENCE) to 109 cm (CREC20%).

Concerning the microstructure of mortars, SEM analysis highlighted an adequate dispersion of cellulose fibres into the mix and a significant presence of binders paste adhered to the surface of fibres. This structure can be translated in a good adhesion strengthened by the rougher surface of fibres. This indicated a well development of the Interfacial Transition Zone (ITZ) between fibres and binder paste.

Concerning the microstructure of mortars, from MIP analysis, the total accessible porosity in mortar raised from 31% for REFERENCE (mortar with no cellulose fibres addition) to 34% for CREC 20% (mortar with addition of 20% by volume of recovered fibre), highlighting two different pore size distribution: unimodal for REFERENCE and bi-modal for CREC 20%. It was observed that the increase of the number of pores and their diameter were originated by a higher amount of cellulose fibres. In particular, as a result of data processing, the second peak of CREC 20% curve shifted to bigger pores values. This is mainly caused by the porous morphology of fibres and slightly by the interaction between fibres and binder paste [30]. In fact, ITZ was optimum as already discussed, but however the interface is present.

Physical and mechanical properties were analysed for each percentage of added fibres (0%, 5%, 10%, 15% and 20% by volume of mortar). On the one hand, the first evident result was the variation in density value which was lower for 20% addition, as expected. In fact, an increase in fibres with consequent porosity increase and binder paste content reduction, gave a decrease in density from 1703 kg m^{-3} for CREC 5% to 1574 kg m^{-3} for CREC 20%.

On the second hand, the higher the fibre addition, the lower the R_c of mortars. For CREC 20%, R_c was the 46% of the reference value (REFERENCE) because the addition of fibres weakens the material, causing more voids [31]. Nevertheless, positive result was found concerning the R_f. In fact, R_f increased gradually (up to 200% of the REFERENCE value for CREC 20%) with the increase of fibres percentage due to the bridging effect that increases the resistance to crack propagation in composites, according to literature references [18][32].

Concerning the hygrometric behaviour, tests on different mortars highlighted an increase in WVP and a reduction of μ (from 11.2 for REFERENCE to 10.2 for CREC 5% and 9.7 for CREC 20%), when fibre addition increased.

As a consequence of this result, adding cellulose fibres, could have a positive impact on mortar behaviour since it allows a good transpirability, drying and high elimination of water vapour in indoor. Further, cellulose fibres addition in mortar implied an increase in MBV, which led to a higher absorption/desorption of water vapour [33].

In order to summarize the results obtained by testing the different properties of mortars, Table 7 compares the performance of mortars with and without fibre addition. For each property, the mortar which had the best behaviour is marked with +.

Table 7. Comparison of mortar properties with and without addition of cellulose fibres.

Properties of mortars	**Mortar with no fibre addition**	**Mortar with fibre addition**
Flexural Strength (R_f)	-	+
Compressive Strength (R_c)	+	-
Lightness	-	+
Water Vapour Permeability	-	+
Moisture Buffering Capacity	-	+

4. Conclusions

In the experimental tests on Falconara and Carbonera wastewater treatment plants the rotating belt filtration was used to improve solids removal efficiency and the recovery of cellulose fibres, derived from toilet paper. Total suspended solids removal efficiencies varied in the range of 11-74% when surface loading rate was 6-108 kg $_{\text{total solids}}$ $m^{-2}h^{-1}$. The obtained separation yields allowed to recover a cellulosic sludge mainly composed by cellulose, hemicellulose, lignin and humic compounds. 26.59 g $_{\text{cellulosic material}}$ m^{-3} was the average value of cellulosic material recovery yield reached.

Then recovered fibres were analyzed and added, at the rate of 0%, 5%, 10%, 15% and 20% by volume, to mortars in order to explore possible benefits in the building sector. As final result of the analysis, the use of recovered fibres in mortars not only allows to add value to a "waste", but also increases sustainability of building productive processes, by reducing the demand of virgin cellulose fibres.

Results showed that these fibres enhance the hygrometric properties of the composites, with consequent positive effects for the human comfort and health. Further, a reduction of compressive strength of mortar resulted from the addition of recovered cellulose fibres, as well as the increase of the flexural strength, thanks to the enhanced adherence of fibres to the binder paste.

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