LCA study for pilot scale production of Cellulose Nano Crystals (CNC) from wood pulp.

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Abstract

Interest in cellulose nanocrystals (CNC)/cellulose nanofibrils (CNF) made from woody biomass has been growing rapidly with close attention from pulp and paper industry, governments, universities, and research institutes. Many new products development with CNCs have been studied intensively. However, little life-cycle analysis (LCA) has been conducted for the environmental impact from these nano-material-containing products and their associated production. Life-cycle analysis can identify environmental "hotspots", which can guide process design changes leading to reductions in the environmental footprint of nanocellulose production. A preliminary LCA was conducted on pilot-scale nanocellulose production at USDA Forest Product Laboratory. The nanocellulose produced at FPL has been tested in many applications, including coatings, microelectronics, high-strength transparent plastic composites, etc. Soon these applications will require the Life Cycle Inventory (LCI)/LCA from the raw material CNC for their new products' environmental impact analysis. Therefore, our LCA studies on nanocellulose production are critical and will be the foundation for further analysis as new products are commercialized.

Keywords: Cellulous nanocrystal (CNC), Life-cycle inventory, life-cycle assessment, commercial production, Nanotechnology
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Introduction

Nanotechnology opens great opportunities for innovation. Unfortunately, the production of many types of nanomaterials can be energy intensive and expensive (Curran et al., 2006, Tsuzuki, 2014), but nanocellulose from forest products have the potential to be processed on an industrial scale at lower cost than other nanomaterials (Jacoby, 2014). Production of nanocellulose and innovative nanocellulose-enhanced products have great potential for growth. According to Jacoby (2014), market forecasters predict by 2020 the nanocellulose market in North America alone will be worth $250 million. Nanocellulose, in form of cellulose nanocrystals (CNC) and cellulose nanofibrils (CNF), are being explored for a wide range of potential applications, including gels and films (Jacoby, 2014), electronics and tissue engineering (Jacoby, 2014, USDA FPL Lab Notes, 2015), concrete building materials, et al. (USDA FPL Lab Notes, 2015).

Uncertainties regarding possible impacts on the environment and human health have surfaced and need to be evaluated. A holistic and comprehensive LCA can provide such information to the public and stake holders. The LCA results can also be used to compare products from emerging nanotechnologies with conventional products and can be used to make informed decisions about further development.

LCA research on cellulose nanomaterials produced from woody biomass is still in its infancy even though considerable research has been done on its production technology and applications. The slow progress in the LCA of nanocellulose is due to a lack of risk-associated information and immaturity of the technology and markets. An LCA study by Li evaluated cellulose nanofibrils (CNF) produced from woody biomass (bleached wood pulp) at a laboratory scale (Li, 2012). Chemical treatments using either the TEMPO (2,2,6,6-tetramethylpiperidine-1-oxyl) or chloracetic acid process, combined with mechanical treatment using either a homogenizer or ultrasound were evaluated. High-energy requirements were reported with sonification more than doubling total energy needs relative to homogenizer methods. Hohenthal et al. (2012) from Finland led a research funded by European Community's 7th Framework Programme reported on the environmental, social, and economic impacts of the cellulose nano-fibrillated production and applications. Their CNF was produced from a dissolving grade of sulfite pulp with chemi-mechanical processes in Sweden using expert's estimates and theoretical data for three different preparation methods and two CNF-imbedded paper products. The analysis is a cradle-to-grave LCA including end-of-life assessment for the CNF enhanced products. The LCA results show the electricity requirement plays a significant role in the global warming impact, greater than raw materials including pulp and chemicals. Another study from Brazil (Figueiredo et al., 2012) of LCA on cellulose nanocrystals used laboratory data for chemical only treating process on coconut fiber and white cotton fiber to produce crystalline nanocellulose. The product system in their study includes fiber processing and electricity and chemical production processes. Their LCA results show that improving the environmental performance of the system needs to focus on improving yield efficiency, reducing energy and water use and recovering lignin and hemicellulose from effluents. The benefits from carrying on an early LCA of the production phase allows for more flexibility and opportunities to improve the processes before implementing commercial process.

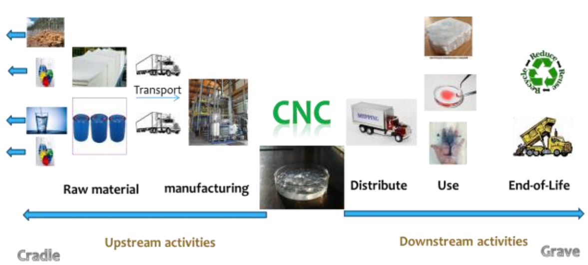
The scarcity of LCA studies on CNC and CNF is due to the lack of mature and conclusive data for production at commercial scale. Only five pilot upscale processes for producing nanocellulose are available in North America: CelluForce and Alberta Innovates in Canada and the Forest Products Laboratory in the United States for traditional cellulose nanocrystals, Kruger in Canada for a mechanical grade of cellulose nanofibrils, and American Process, Incorporated, with a pilot line using a different chemical process method that produces both rod-shaped particles or fibrils. The USDA Forest Product Laboratory (FPL) pilot line has production capacity of 25 kg CNC per batch or 5 kg CNF per batch (Reiner and Rudie, 2013). Data collected for this LCA study are specific and precise for the FPL process.

Goal and Scope

This study is intended to collect the life-cycle inventory data for CNC produced from FPL's newly developed pilot plant, and conduct a preliminary life-cycle assessment for nanocellulose from woody biomass. The life-cycle analysis of this study can help to understand the environmental impacts of the CNC fabrication process and identify the hotspots in the CNC production chain. Using the LCA results from this pilot line CNC production can also guide the design of commercial scale CNC production to minimize energy and environmental impacts.

The scope of this study covers the raw materials extraction and production, including bleached kraft pulp from wood chips, chemicals such as sulfuric acid and alkali, transportation of these raw materials, and the CNC pilot plant operation including heat and electrical. It is a cradle-to-gate LCA study, as the gate being the CNC product produced before distributing for use as shown in Figure 1.

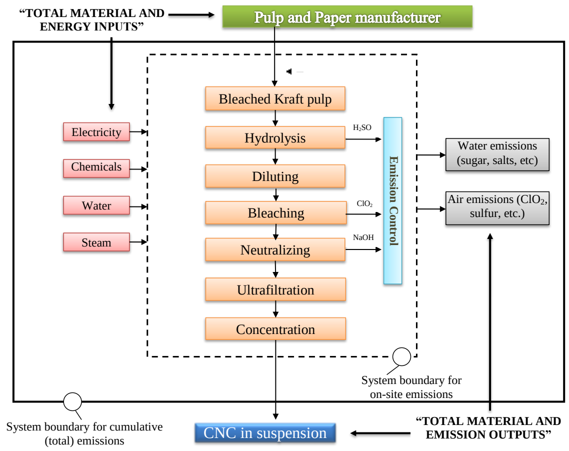
Figure 1: Cellulose nanocrystals (CNCs) cradle-to-grave life-cycle demonstration



Functional Unit –For this study, 1 kg of CNC produced from the pilot acid hydrolysis process is defined as the functional unit for the impact analysis.

System boundary – Defining the system boundary selects the unit processes to be included in the system. Based on our goal to conduct a preliminary LCA analysis for CNC produced by the FPL pilot plant, the system boundary was drawn to include the upstream material resources and handling, chemicals transportation, and the CNC manufacturing (Fig 1). Downstream activities including product distribution, applications and end-of-life treatment are not considered. Figure 2 shows the system boundary for the cradle-to-gate LCA analysis in this study. The cumulative system boundary includes both on- and off-site emissions for all material and energy consumed. The onsite emissions include the processes within the dotted line (Fig 2). The off-site emissions include the grid electricity production, chemicals purchased, and water and steam produced off-site but consumed onsite. Fuel and electricity use for the upstream feedstock and chemicals production are estimated. The CNC manufacturing processes are estimated in detail based on the FPL production data recorded and included in the cumulative boundary (solid line) to calculate the total emissions.

Figure 2: System boundary



Method

Acid hydrolysis was developed in the 1940s and remains a major process to produce stable colloid suspensions of cellulose nanocrystals with negative charges. The process is low yield about 50% starting with bleached wood pulp. The pilot plant at FPL consists of five reactors to produce both CNC by acid hydrolysis and TEMPO-oxidized CNF. A membrane filtration system with 7.5 m^2 of 200,000 MW cutoff membrane is used for purification (diafiltration) and concentration of the CNC aqueous product. An onsite reverse osmosis (RO) system is required to produce purified water needed for this production line. Figure 3 shows the end view of the pilot line with ultrafiltration system displayed in the foreground.

Materials

The pilot-scale line CNC production starts with 50kg machine-dried pre-hydrolysis kraft rayon-grade dissolving pulp. Dissolving pulps are nearly pure cellulose

Figure 3: CNC/CNF Pilot-scale production line at USDA Forest Products Laboratory in Madison, WI.

and provide a higher yield of the CNC product. Shredded into strips, the pulp is packed into the 400-L glass-lined reactor and placed under a nitrogen atmosphere. Small amount of nitrogen was introduced into the hydrolysis reactor tank to purge the oxygen which may cause oxidization of cellulose. Such small amount of nitrogen was neglected in the inputs of the model. Warm water is circulated through the hydrolysis reactor water jacket to keep the temperature at 45°C. 300 L of 64% wt sulfuric acid solution is heated to 45°C in a second 400 L reactor, then transferred to the pulp reactor where it is sprayed over the top of the shredded pulp. The hydrolysis reactor mixer is turned on and the reaction held at 45° C for 90 minutes. The CNC reaction is quenched by transferring the suspension to a 6000 L glass lined reactor containing 1200 L of RO water for diluting. About 1 kg of sodium chlorite is added to the acidic suspension and mixed for about 30 minutes; the chlorine dioxide that forms bleaches the CNC suspension that was discolored by sugar degradation during the hydrolysis reaction. Then 3000 L of 10% NaOH solution is added to neutralize the acid, taking about 0.75 hours. Finally, about 2 L of 4% hypochlorite solution is added to further remove color. The aqueous suspension is then transferred to the ultrafiltration system to remove the byproducts of the reaction, primarily glucose and sodium sulfate. CNCs are circulated through the tubular ultrafiltration system, where the salt and sugar pass through the membrane while CNCs are retained. RO water is added as needed to maintain the CNC concentration at 1 wt%. This diafiltration requires about 36 hours of dilution and filtration and about 21,000 L of RO water. The colloidal CNC suspension is filtered using a 20-µm polypropylene cartridge-style filter to remove dirt introduced during the transfer and processing, and the membrane ultrafiltration system is used again to concentrate to 12 wt% CNC in the final suspension product. Total running time for the ultrafiltration system is estimated at 40 hours. The yield for CNC produced from the dissolving pulp is approximately at 50%.

Results and Discussion

FPL's CNC line produces 25 kg CNC per batch from 50- kg oven-dry dissolving pulp. The production data were collected from this batch operation and inputs and outputs for modeling the life-cycle inventory are summarized in Table 1.

Life-cycle analysis software SimaPro 8.0 was used to model the process. Feedstock pulp was purchased from a special supplier with transportation included in the SimaPro modeling. Transportation of the purchased pulp and chemicals was included (Table 1). A mass balance was calculated based on the raw materials and chemicals used in the processes and output products including emissions to air and water. Reverse osmosis (RO) water was processed onsite to supply the production. Total amount of RO water and electricity use were determined based on the operation and engineered design.

Dissolving pulp was chosen for this process because of its higher cellulose content which provides a higher yield of CNC and produces less coloring of the product from degradation of hemicellulose sugars. However, in the SimaPro no exact dissolving pulp can be found in the US LCI or US Ecoinvent database. A process-closed bleached kraft pulp was chosen in this preliminary modeling to approximate the starting pulp.

Life-Cycle Inventory (LCI) –

Life-cycle inventory for 1-kg CNC produced from FPL's scale-up production line was completed from the SimaPro model with US LCI database and US Ecoinvent database.

The cradle-to-gate cumulative energy for producing 1 kg of CNC from LCI result is estimated as 992.7 MJ as shown in Tables 2 and 3. For Li's lab scale CNC product using mechanical and chemical combined process, cumulative energy was estimated from 3470 to 17610 MJ/kg (2013). Another lab scale CNC process estimate from Figueiredo et.al (2012) resulted in a cumulative energy estimate range from1800 to 15943 MJ/kg.

Table 1: LCA Input-Output for producing 1 kg CNC with the FPL pilot scale-up production line

Stage	Output	Amount	Unit
CNC Feedstock	CNC shredded pulp (~10%MC)	55.56	kg
	waste (pulp waste)	0.05	kg
	Input		
	Bleached kraft dissolving pulp	55.61	kg
	feedstock transport	1400	km
CNC Hydrolysis reaction	**Output**	**Amount**	**Unit**
	Hydrolized CNC	25	kg
	Sugar	25	kg
	Input		
	CNC shredded pulp	55.56	kg
	Sulfuric Acid	296	kg
	Steam	21.8	kg
	Electricity use in mixing	3.357	kWh
	Electricity use for pump	11.19	kWh
Dilution, Bleaching and Neutralization	**Output**	**Amount**	**Unit**
	Neutralized CNC	25	kg
	ClO2	0.2	kg
	Input		
	Hydrolized CNC	25	kg
	RO water	5735	liter
	Sodium Hydroxide NaOH	241.4	kg
	Sodium Chlorite NaClO2	1	kg
	Clorox Sodium Hypochloride	*0.111*	*kg*
	Electricity use in mixing	6.994	kWh
Ultrafiltration	**Output**	**Amount**	**Unit**
	CNC product	25	kg
	waste water	26552	liter
	NaCl	0.342	kg
	Na_2SO4	427.8	kg
	Input		
	Neutralized CNC	25	kg
	RO water	21000	liter
	Electricity use for RO water	14.32	kWh
	Electricity use for pump	268.56	kWh

The higher energy result was due to the scale of their production for CNC product and limited access to the advanced nanotechnologies. Table 2 distributes cumulative energy into 4 categories and Table 3 reports the energy consumption contributed by different components in the production. About 88% of total energy was consumed from non-renewable sources and only 12% from renewable sources including biomass, solar, wind and hydropower (Table 2). Most of the energy (about 65%) was used in producing chemicals, such as sodium hydroxide, sulfuric acid, etc. Feedstock pulp and electricity only contributed to 16% and 15% of the total energy (Table 3, Fig 4).

Table 2: Cumulative energy consumption for every kg of CNC produced at FPL showed in different categories

Substance	Unit	Total	(%)
Non-renewable fossil	MJ	826.56	83.26%
Non-renewable Nuclear	MJ	45.91	4.63%
Renewable, biomass	MJ	112.74	11.36%
Renewable (solar, wind and hydropower)	MJ	7.48	0.75%
Total		**992.7**	**100%**

Emissions to the air and water for producing 1 kg CNC with FPL's processes are summarized in Tables 4 and 5 from the LCI results. Most of the air emissions and water emissions are from producing sodium hydroxide and feedstock dissolving pulp.

Sodium hydroxide was used in this study for neutralizing the acid solutions after the acid hydrolysis reaction. Larger commercial processes will have to recover sulfuric acid and that will significantly reduce the amount of sodium hydroxide needed to neutralize acid.

Table 3: Energy consumed for each input component of producing 1 kg CNC

Produce 1 kg CNC	MJ	%
Total cumulate energy	**992.7**	**100%**
Feedstock - bleached pulp	158.21	15.94
Total transportation	6.47	0.65
Sulphuric acid	26.22	2.64
RO water	24.17	2.44
Sodium chloride	0.093	0.01
Sodium hydroxide	620.94	62.55
Sodium hypochlorite	1.49	0.15
FPL process steam	7.32	0.74
FPL process electricity	147.68	14.88

Figure 4: Contribution to the total cumulative energy from each input component

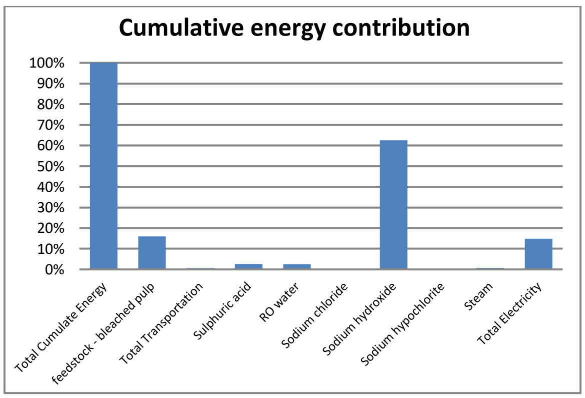


Table 6 illustrates the energy intensity and ultimate tensile strength of a range of materials (Kuehr et al. 2003) compared to CNC. The energy intensity is expressed in energy use per physical unit of output for each of these commodities. The energy intensity (or embodied energy) values provided in the Table 6 are defined as the energy required to produce one unit mass of material from its raw form (Gutowski et al., 2013). Cellulose as a structure material is extremely strong, with a theoretical modulus of around 250 GPa and a specific tensile strength of about 5200 MPa. This value is about 10 times that of steel and 115 times of aluminum comparing the values showed in Table 6. While the estimated energy intensity of CNC produced from FPL's pilot line is 993 MJ/kg, which is about 17 times that of steel and four times of aluminum as compared to the values in Table 6.

Table 6: Energy intensity and ultimate Tensile strength for a range of materials

Material	Energy intensity of materials (MJ/kg)	Ultimate tensile strength (MPa)
Steel	59	500
Copper	94	210
Silver	1570	170
Aluminum	214	45
Lead	54	12
Nickel	340	175
Gold	84000	100
CNC	**993**	**5200**

Life-Cycle Assessment –

Environmental impacts are integrated in 10 categories by SimaPro using TRACI (The Tool for the Reduction and Assessment of Chemical and other environmental Impacts) method (Bare 2011), and presented in Table 7. The Life Cycle Impact Assessment (LCIA) links the life-cycle inventory data with each environmental impact and provides general, quantifiable indicators for potential environmental impacts. Global warming potential is a measure of the contribution to climate change from a process. Global warming impact calculates the total emissions in the reference unit of CO_2 equivalents. It includes real CO_2 emission and methane and nitrous oxide emissions referenced to CO_2. Global warming potential is among the most closely followed environmental impact by the environment and climate change studies (Curran et al 2006, Figueiredo et al 2012, Hohenthal et al 2012, Li 2012, Li et al 2013) . Ozone depletion measures the total ozone forming chemicals in the stratosphere including chloroflurocarbons, etc. Smog calculates total substances that can be photo-chemically oxidized. Acidification measures total hydrogen ion (H+) for released sulfur oxides, nitrogen oxides, hydrochloric acid, etc. It is a potential of a chemical emission to acidify ecosystems. Eutrophication measures total substances that contain available nitrogen or phosphorus, usually refers to aquatic eutrophication. It occurs when excessive amounts of nutrients reach freshwater system or oceans. Human health and wildlife impact related indicators include carcinogenics, non-carcinogenics, respiratory effects, and ecotoxicity. These indicators measure the substance or particulars that are emitted to the air or water affecting human health or wildlife system. Fossil depletion measures the depletion of non-renewable resources. It is an important metric for sustainability.

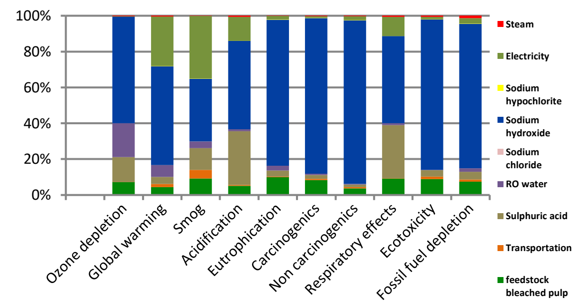
Table 7: LCA for producing 1 kg CNC with FPL's pilot production process

Impact category	Unit(/kg CNC)	Total
Ozone depletion	kg CFC-11 eq	1.11E-06
Global warming	kg CO2 eq	29.64
Smog	kg O3 eq	1.68
Acidification	kg SO2 eq	0.54
Eutrophication	kg N eq	0.05
Carcinogenics	CTUh	8.25E-07
Non carcinogenics	CTUh	8.71E-06
Respiratory effects	kg PM2.5 eq	0.03
Ecotoxicity	CTUe	96.81
Fossil fuel depletion	MJ surplus	72.11

Environmental performance contributions are shown in Figure 5 for each impact category for the different chemicals, feedstock pulp, transportation, RO water, electricity and steam use.

The highest environmental impact is from sodium hydroxide production in all categories. For global warming impacts (GWI), 55% of CO_2 equivalent emissions come from sodium hydroxide, 28% come from electricity, the second highest contribution to the GWI after sodium hydroxide.

Figure 5: Percentage contribution from each activity to the impact categories from LCA



Conclusion

From the LCA results discussed above, the impacts from sodium hydroxide are the dominant contribution in the cradle-to-gate LCA. Sodium hydroxide was used to neutralize the acid solution after the hydrolysis reaction. So the solution will be able to drain into the waste stream without any further treatment. This is specially designed for the FPL's pilot production based on consideration of easy waste stream management. In a commercial process such use of chemicals would not be ideal. An ideal design, which is proposed by FPL for commercial scale production (Houtman and Rudie 2015), is to recycle the acid solution for reuse. This will eliminate all but about 10% of the sodium hydroxide and reduce energy and all other LCA impacts for this process.

For cellulose-based nanotechnology to move forward, production scalability and sustainability are necessary for a study. The

LCA and estimated environmental impacts from this research guide research and process design priorities for the next scale and commercial scale CNC plants in order to attain sustainable operations.

LCA helps to identify the potential risks associated with nanomatierials production and nano-enhanced products. These concerns need to be addressed in the development efforts to build larger scale production facilities.

Table 4: Air emissions from producing 1 kg CNC by FPL pilot scale-up production line

Substance	Unit	Total	feedstock pulp	Transportation	Sulphuric acid	RO water	Sodium chloride	Sodium hydroxide	Sodium hypochlorite	Transportation	total Electricity	Steam
Carbon dioxide, fossil	kg	23.88973	4.4%	1.2%	4.6%	0.0%	0.0%	55.9%	0.1%	0.8%	32.7%	0.3%
Carbon dioxide, biogenic	kg	5.409814	93.3%	0.0%	0.1%	0.0%	0.0%	5.0%	0.0%	0.0%	1.6%	0.0%
Carbon dioxide	kg	1.880644	0.0%	0.0%	0.0%	100.0%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
Carbon monoxide, fossil	kg	0.013128	17.7%	11.1%	3.9%	0.0%	0.1%	40.1%	0.1%	7.7%	19.3%	0.1%
Sulfur dioxide	kg	0.484262	4.6%	0.0%	32.2%	0.7%	0.0%	50.6%	0.1%	0.0%	10.9%	0.7%
Methane, fossil	kg	0.12486	7.6%	0.0%	2.5%	0.0%	0.0%	88.1%	0.2%	0.0%	0.3%	1.4%
Methane	kg	0.019714	0.6%	1.7%	0.2%	14.9%	0.1%	6.9%	0.0%	1.2%	74.3%	0.1%
Nitrogen oxides	kg	0.063959	9.6%	3.0%	12.8%	0.0%	0.0%	36.7%	0.1%	2.2%	35.5%	0.1%
NMVOC, non-methane volatile organic	kg	0.010159	14.0%	1.5%	4.7%	1.3%	0.0%	71.8%	0.2%	1.1%	4.3%	1.1%
Particulates, < 2.5 um	kg	0.002529	61.7%	0.0%	7.7%	3.7%	0.0%	26.5%	0.1%	0.0%	0.0%	0.3%
Particulates, > 2.5 um, and < 10um	kg	0.000666	2.7%	5.1%	3.6%	16.7%	0.2%	9.8%	0.1%	4.2%	57.7%	0.1%
Particulates, > 10 um	kg	0.010108	3.8%	0.0%	2.3%	0.0%	0.0%	93.8%	0.2%	0.0%	0.0%	0.0%

Table 5: Water emissions from producing 1 kg CNC by FPL pilot scale-up production line

Substance	Unit	Total	feedstock pulp	Transportat ion	Sulphuric acid	RO water	Sodium chloride	Sodium hydroxide	Sodium hypochlorite	Steam	total Electricity
Magnesium	kg	17.112	0.0%	0.0%	0.0%	0.0%	0.00%	0%	0.00%	0.00%	0.0%
Sulfate	kg	4.595	6.9%	0.0%	4.3%	29.4%	0.00%	59%	0.11%	0.02%	0.0%
Lithium	kg	1.802	7.2%	1.3%	3.3%	0.0%	0.01%	84%	0.22%	1.45%	2.0%
COD, Chemical Oxygen Demand	kg	1.871	5.9%	1.0%	2.0%	13.3%	0.01%	74%	0.95%	1.14%	1.5%
Magnesium	kg	1.064	0.0%	0.0%	0.0%	0.0%	0.00%	0%	0.00%	0.00%	0.0%
TOC, Total Organic Carbon	kg	0.618	5.7%	0.8%	2.1%	23.8%	0.01%	65%	0.16%	0.98%	1.3%
COD, Chemical Oxygen Demand	kg	0.445	3.7%	0.0%	3.0%	0.5%	0.00%	93%	0.17%	0.01%	0.0%
Aluminium	kg	0.256	6.6%	0.6%	2.0%	5.4%	0.01%	83%	0.19%	0.74%	1.0%
Chloride	kg	0.076	5.3%	0.4%	2.2%	0.0%	0.00%	91%	0.19%	0.49%	0.7%
COD, Chemical Oxygen Demand	kg	0.042	7.3%	0.1%	2.1%	0.0%	0.01%	87%	0.23%	1.49%	1.4%
Iron	kg	0.070	52.6%	0.3%	4.6%	23.7%	0.00%	18%	0.05%	0.32%	0.3%
Sodium	kg	0.037	5.4%	0.0%	2.3%	0.0%	0.00%	92%	0.17%	0.01%	0.0%

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