Egypt. J. Chem. **Vol. 68**, No. 10 pp. 659 - 667 (2025)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Minimize environmental impact of dairy production through improve the life cycle. Part 2: "Recovery of Proteins and Nutritional Components from Dairy Whey Waste via Spray Drying Technology"

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Mohamed O. El-Tawabty [1]; Alaa A. M. Samn [2]; Mohamed H. Ghanem[2]; Noura A.[3]; Enas M. Abou Taleb[4]

[1] 6th of October City Authority, New Urban Communities Authority, Ministry of Housing and Utilities, P.O. 12573, Giza, Egypt
[2] Zoology Department, Faculty of Science, Al-Azher University, P.O. 11884, Nasr City Cairo, Egypt
[3] Animal Physiology Research, Desert Research Center, P.O. 11753, Cairo, Egypt
[4] National Research Centre, Water Pollution Research Department, El Buhous St., P.O. 12622, Dokki, Giza, Egypt

Abstract

Mechanical wastewater from dairy plants contains high concentrations of natural toxins that are harmful to the environment. Dairy plant pollution is characterized by high levels of natural substances, which represent a major source of chemical oxygen demand, biological oxygen demand, total suspended solids, and total organic carbon. Using spray-drying technology, this study formulated a novel strategy for extracting protein and nutrients from dairy plant sludge. A comparison was made between conventional organic processing and protein drying using animal feed by-products as an alternative to conventional proteins. The process of spray drying represents an established physicochemical methodology for the transformation of liquid-state solutions into fine powders, and droplets are heated with hot air, which leads to rapid water evaporation. Thus, the spray-drying method offers low waste with a conservative yield, resulting in the removal of contaminants in large mechanical filter units. The results showed that the treated effluent of the dry system complies with wastewater limits. The dry system effectively removes chemical oxygen demand, biological oxygen demand, total suspended solids, and total organic carbon. Furthermore, the dry milk powder was analyzed and evaluated, and it met international standard limits for buttermilk powder. Animals reuse the buttermilk powder due to its high protein content. The results of the analysis of the buttermilk powder showed the presence of protein, lactose, starch, fat, and moisture (34-37, 49.5-52, 0.6-8.6, 0.6-1.25, and 3.0-4.0%), each separately.
Keyword: Dairy; Wastewater; Spray Drying Method; Buttermilk; Protein

1. Introduction:

Water plays a vital role in the global economy. The release of untreated, or even partially treated, wastewater poses a threat to the environment and water resources (1,2). The amount of toxins has increased as a result of industrialization, in addition to the increase in human activity (3,4). It is essential to maintain water quality standards set by appropriate controls and to eliminate harmful substances from wastewater that have recently leaked into the environment (5). The main reason behind the growing interest in reusing treated wastewater is the increasing demand for water resources (6,7).

Manufacturing is largely dependent on environmental pollution, especially of water, as lakes are flooded with massive amounts of toxic substances. Heavy metals reach dangerous levels compared to other toxic substances (8,9). Milk production must be based on scientific studies, as it is one of the most important human foods. The dairy industry includes the conversion of raw milk into pasteurized and sour milk, yogurt, soft cheese, cottage cheese, cream and butter products, ice cream, milk, milk powders, lactose, condensed milk, and various types of desserts. Various sources of pollution in the dairy industry are classified (10).

Whey, which makes up 20% of milk proteins, is produced as a by-product of the dairy industry. Whey is a rich source of numerous bioactive compounds and contains numerous essential nutritional supplements (11,12). Whey is available in several forms, including liquid whey from cheesemaking, whey protein concentrates, or whey extracts(13, 14).

There are many methods for treating wastewater such as activated carbon neama at al, corn cob adsorbent fatehy et al, nanomaterials Huusein et al, constructed wetland system Hussein et al, Abdel-Shafy et al, agro-waste materials El-Khateeb et.

*Corresponding author e-mail: mohamedosama25@hotmail.com.; (Mohamed O. El-Tawabty).
Receive Date: 18 April 2025, Revise Date: 11 June 2025, Accept Date: 15 June 2025
DOI: 10.21608/ejchem.2025.376829.11621

al, chemical coagulants Hussein et al, ultrafiltration, nano-filtration, reverse osmosis, and thermal concentration can be used to concentrate proteins in the dairy industry (15, 16).

In dairy plants, product cleaning is the primary source of wastewater. Typically, wash water is generated from the production line cleaning process, and washing processes include pre-cleaning, main cleaning, neutralization, or disinfection of wash water (17). The wastewater generated by these washing processes contains a significant amount of protein from commercial cleaning products.

Dairy plants are under constant pressure due to the ever-increasing enforcement of environmental regulations (18). However, these wastes contain high levels of protein and therefore have potential high nutritional value (19, 20, 21).

The manufacturing process of milk powder involves multiple sequential phases of thermal and mechanical treatment such as initial phase separation, thermal processing, thermal pre-conditioning, aqueous phase reduction, structural stabilization (WMP specific), rheological modification, and Dehydration (22).

The process of spray drying represents an established physicochemical methodology for the transformation of liquid-state solutions into discrete solid particulates. This technique has found extensive application in dairy processing operations, with particular prominence in whey treatment protocols.

Spray drying is a method used in most industrial processes, especially in the pharmaceutical, food, and chemical industries, to produce fine powders from a fluid material for further use.

The process is generally applicable to liquids fed into the arrays, which disperse the feed material into a mist of small droplets (23). Droplets are heated with hot air, which leads to rapid water evaporation, such that dry droplets are formed partway through the drying vessel and allow a constant drop size for the remaining drying process. After water evaporation, particles reach a glass transition temperature where solid particles are preserved, preventing excess softening by the expelled solvent. The powders can be discharged from the conical portion, which contains particles, by way of containers. The value of spray drying technology arises from its effectiveness in drying liquids into powders and its ability to ensure good shelf stability of products in comparison to freeze-drying technology. Spray drying technology has several advantages for developing powder products, including controlled processing conditions, simple operations, cost-effectiveness, and industrial suitability in contrast to lyophilizers (24, 25). Using spray-drying technology, which is the most common operation to concentrate milk and convert it to other products, has not been applied to concentrate proteins and extract these valuable ingredients from dairy liquid residues, so we aimed to carry out this study (26, 27, 28).

The discharge of whey effluent in the 6^{th} of October Industrial City presents significant environmental implications that require immediate intervention. Previous research indicates minimal implementation of whey waste management protocols (29). Whey effluent is characterized by high biological oxygen demand (BOD_5) and substantial volumes of process wastewater with elevated organic loading rates. The variable composition and quantity of this wastewater stream results in inconsistent total suspended solids (TSS) and organic loading patterns within treatment facilities.

This study primarily focuses on the recovery of nutritional values present in the wastewater of a plant where it processes milk and milk by-products. It is proposed to reveal the potential protein and mineral content in the dried concentrated form of these losses and re-evaluate whether they can be used again. Along with this evaluation, it is also aimed as a determining study, revealing the efficiency of spray drying technology, which is used in drying to evaluate the amount of solid mass using the data of the amount of concentrated mass to carry the same dry substance. In this study, whey waste product is characterized by a variety of pH values in chemical oxygen demand (COD), BOD_5, TSS, total organic carbon (TOC), and lactose. Considering all, this study aims to estimate the polluting load created by dairy industrial effluents in one of the foremost critical industrial zones in Egypt, evaluate the system of the treatment of whey waste product using a heating system, and highlight the transformation of raw milk into products like consumer milk, butter, and cheese.

2. Material and Methods

2.1. Selection and specification of the studied station

The study area is stored in the industrial zone of the 6thof October City in the governorate The study area consists of eleven industrial zones in the 6th of October City, Giza Governorate (Egypt), starting from the first zone to the eleventh zone, and includes all industries, especially the dairy industry and all its derivatives. This research deals with the industrial zone in the sample. Table (1) shows a description of the industrial zones, and based on it, the specifications of the studied area (7) were collected (7).

2.2 Samples collection

Effluent samples containing whey by-products were systematically collected from a dairy processing facility situated in Egypt's 6^{th} of October Industrial Zone from 11 areas starting from the first zone to the 11^{th} zone with all industries. The sampling protocol involved weekly collection of eleven distinct wastewater specimens over a 180-day period to ensure comprehensive temporal representation of effluent variability.

Table 1: The description of industrial zones

Sample Code	Sample Name	Location		Product Description
		Latitudes N	Latitudes E	
SP 1	1st industrial zone	29 56 02	30 54 18	Whole milk-Whey-Curd-Butter-Cheese- Milk powder-Cream-Yogurt
SP 2	2nd industrial zone	29 56 02	30 53 51	
SP 3	3rd industrial zone	29 55 26	30 53 05	
SP 4	4th industrial zone	29 56 38	30 52 47	
SP 5	5th industrial zone	29 54 12	30 54 17	Yogurt- Cheese -Whole milk
SP 6	6th industrial zone	29 55 17	30 52 26	Cheese-Whole milk
SP 7	3rd industrial zone extension.	29 55 13	30 52 57	Whole milk-Whey-Curd-Cheese-Milk powder
SP 8	4th industrial zone extension.	29 56 57	30 51 21	Whole milk-Whey-Curd-Cheese-Cream-Yogurt
SP 9	6th of industrial zone extension	29 55 09	30 52 35	Whole milk-Whey Cream
SP 10	The storage zone (1)	29 55 45	30 51 17	Whey-Curd- Butter -Cheese
SP 11	The storage zone (2)	29 54 57	30 52 01	

Chemical and physical analyses were conducted to evaluate compliance with regulatory parameters established by Law 92/1963 and ministerial decree 44/2000, specifically pertaining to public sewerage system discharge standards. Quantitative analysis of the industrial zone's dairy production revealed the following daily output metrics: 333 metric tons of whole milk, 89 tons of whey, 130 tons of curd, 57 tons of butter, 96.25 tons of cheese, 75 tons of milk powder, 101 tons of cream, and 71 tons of yogurt, culminating in an aggregate production volume of 952.25 tons per day. The facility generates approximately 33,400 m^3 of effluent daily, characterized by elevated organic loading, which is subsequently channeled into municipal treatment infrastructure. This huge production discharges a huge amount of highly loaded organic contamination in municipal wastewater with a total flow that reaches 33400 m^3/day (Figure 1) showing the relationship between the product of dairy and the types of samples **[7].**

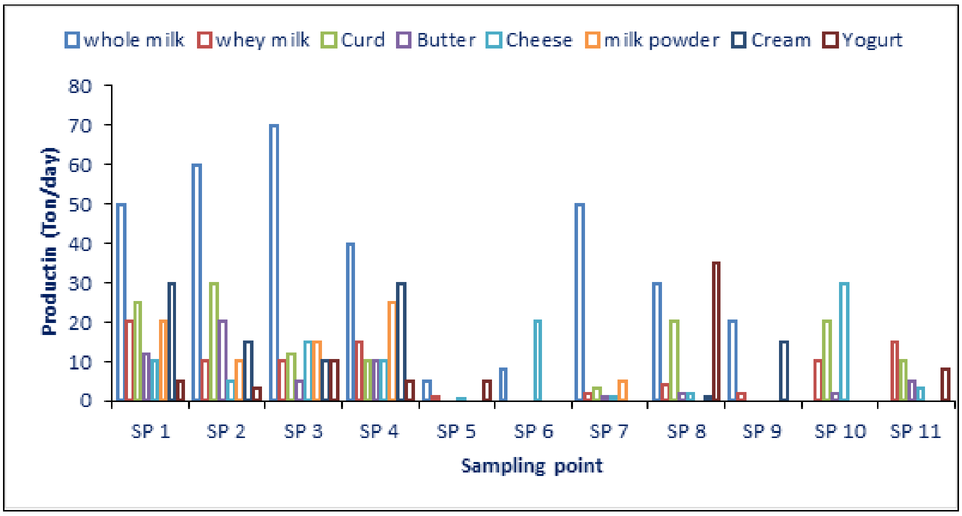

Figure 1: The relationship between the dairy product and types of samples [7]

2.3. Procedure of Drying System Technique

The drying system consists of a mixing tank with a capacity of 250 mL in which the raw liquid was well mixed at 50 rpm. The well-mixed liquid was then transferred by a peristaltic pump with a flow rate of 70 mL/h to the evaporating unit in which a flow of hot air is passing through the unit. The heater was adjusted at a temperature between 50 and 70 ^{o}C and the inlet air at 200 liters per minute. The water vapor is then collected and evaporated through a vacuum pump while the dry powder is collected in a polyproline tank with a volume of 250 mL. the graphical abstract shows a schematic diagram of the system used for the treatment of the whey waste product.

2.4 Analysis of the whey waste product in the studied area and product

2.4.1 Chemical analysis

The analyses of parameters TSS, COD, BOD_5, and TOC were conducted according to the standard methods for the examination of water and wastewater. All analyses were conducted in triplicate runs according to the standard method for water and wastewater examination APHA. 23rd Edition).(30)

2.4.2 Analysis of milk

All analyses were conducted in triplicate runs and all lab experiments were conducted by certified ISO17025 (according to the Association of Official Analytical Chemists (AOAC), 2000 (31). These analyses of parameters were carried out: estimation of the percentage of fat, protein estimation, casino estimation, lactose rating, and ash estimation.

3. Results and Discussion

3.1 Whey waste product characterization and its treatment processes

The effluent characteristics from dairy processing operation at different samples point as illustrated in Figure (2), the milk powder processing protocol encompasses thermal pretreatment followed by solids concentration via evaporative processes and subsequent spray drying methodology. The experimental design incorporated three distinct expired dairy matrices - milk, yogurt, and cheese - selected for their elevated nitrogenous content and nutritional parameters. The separated pollutant fraction, characterized by high lipid concentrations, was allocated for animal nutrition studies. The composite dried milk powder, derived from these expired dairy matrices, underwent mechanical grinding for homogenization. Quantitative analysis revealed mean concentrations for COD, BOD_5, TSS and TOC of 4048 mg/L, 1983 mg/L, 1393 mg/L and 903 mg/L, respectively.

Figure (3) presents the distribution of pollutant concentrations derived from whey waste products across various industrial samples. The whey waste fraction exhibited significant organic loading, predominantly composed of milk constituents, manifesting in elevated levels of COD, BOD_5, TSS, and TOC. Characterization revealed high concentrations of dissolved organic components, including whey proteins, lactose, and lipids (32). Additionally, the decomposition of organic constituents resulted in malodorous emissions, creating adverse environmental conditions for proximate populations (33). Lipid constituents, primarily comprising animal and vegetable fatty matter and petroleum-derived hydrocarbons, demonstrate significant interference with wastewater treatment systems. These interferences are compounded by sulfur compounds and organic dye materials. Furthermore, elevated lactose concentrations in industrial effluent exhibited detrimental effects on biological treatment efficacy. While lactose maintains significant value in pharmaceutical applications, it predominantly exists as a waste product in dairy processing. Implementation of lactose extraction and separation methodologies from wastewater streams presents dual benefits: reduced treatment costs and potential byproduct utilization. Research initiatives focusing on lactose separation and recovery from dairy sector wastewater have been conducted to mitigate organic loading in sewage systems (34). Significantly, approximately 90% of dairy industry BOD_5 and COD loading is attributable to lactose concentration. Cheese whey wastewater demonstrates notably elevated BOD_5 levels and organic matter content, necessitating specialized treatment considerations.

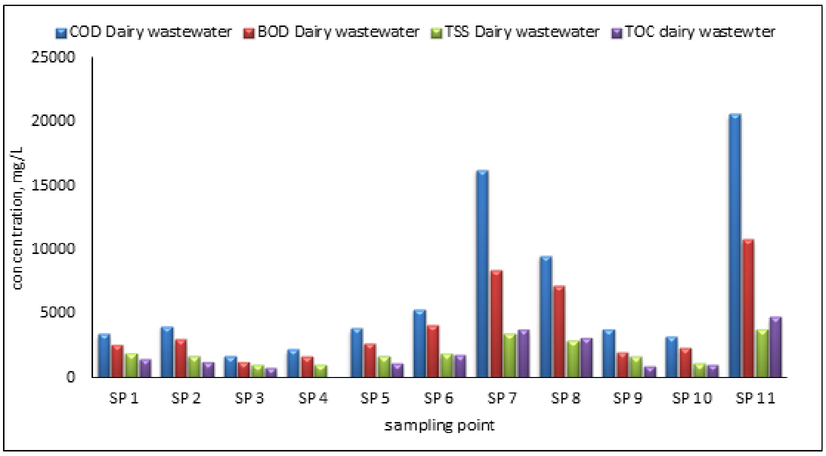

Figure 2: Pollutant concentrations in dairy wastewater at different sampling points.

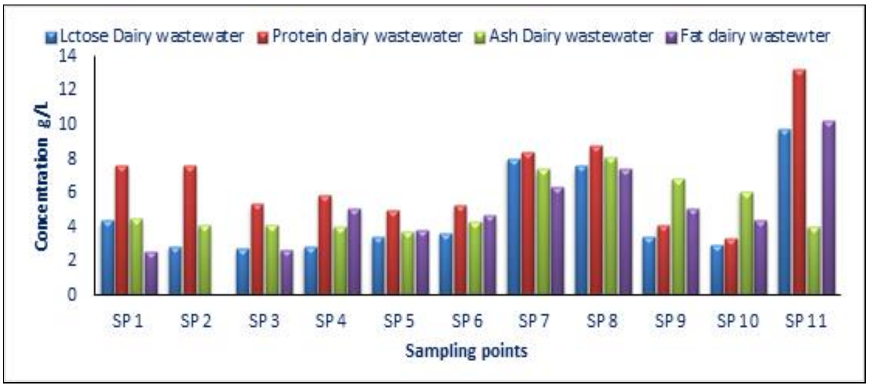

Figure 3: Constituent of different compounds in dairy wastewater

Figure (4) shows the variation of milk content produced from Whey waste product in different Industrial zones. The milk protein comprises two major classes, the casein and proteins. The caseins represent (6.625 g/L proteins), 3.221 g/L lactose, 4.18 g/L ash, and 3.14 g/L fat. However, even in the absence of a major protein, this fraction remains very polluting to the environment, considering the high BOD_5 of lactose, the dominant compound.

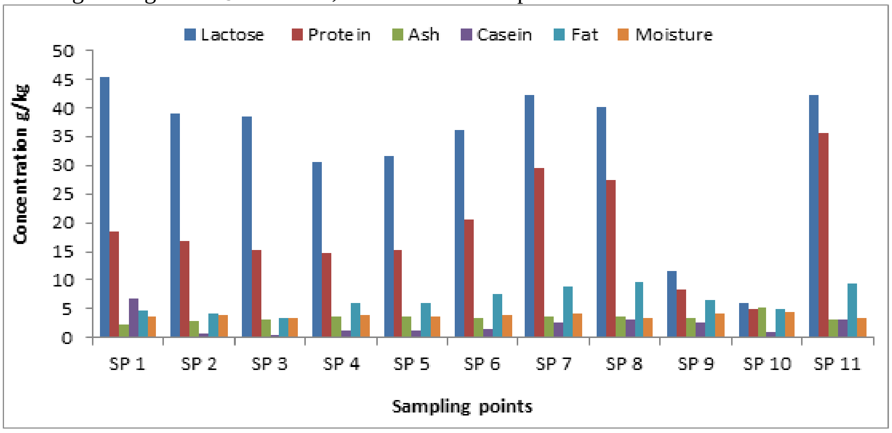

Figure 4: Variation of milk content produced from Whey after spray drying

3.2 The shelf life, packaging of buttermilk powder, and using as animal feed

The results declared that the maximum value of contents was observed in dry milk powder samples as follows: fat (0.6 – 1.25%), protein (34-37%), lactose, (49.5- 52), ash (0.6 – 8.6%), and moisture (3.0 – 4.0%). The microbiological analysis of dry milk powder samples are as follows: coliform, E. coli, salmonella, listeria, coagulase, and staphylococci and their results are as such: < 10mg, negative, negative, negative, positive, and negative, respectively. The physical characterization of dry milk powder is shown in Table (2).

The results indicate the identification of butter milk powder, which was produced in accordance with the accepted standards of good manufacturing practices and in accordance with the legislation of the manufacturing country, and each package must be labeled by the manufacturer name (MO-23), manufacturing location (6^{th} of October City), and the date of manufacturing (2023). The treated and processed product is packed in bags with a polyethylene container and is stored under cool dry conditions (under 25 ^{0}C).

The presented data in Table (2) shows the byproduct of the dry system used in animal feed due to the ingredients of nutritional value that it contains, i.e., lactose and protein are considered among the best content in animal feed. Several studies were conducted on the recovery of protein, lactose, and fat from dairy industry wastewater and reusing them as animal feed. Typically, when comparing dry milk powder in this study with other feed such as whey, it is evident that the whey contains, at least, lactose (48.0%), protein (0.1%), ash (0.1–0.3%), moisture (4.0–5.5%) and is free from fat.

Table 2: Physicochemical and microbiological analysis of buttermilk

Analysis	**Dry powder milk (in this study)**	international standard limits for butter milk powder (Al-Hilphy AR **et al) (32)**
pH	6.4 - 6.6	6.3 - 6.8
Flavor/ Clean	Pleasing dairy flavor	Free from any taints or rancidity.
Color	White to light cream color	a creamy-white
Solubility	Index 1.0 ml	1.25 ml
Titra-table acidity %	0.14 – 0.15	0.18% as Lactic Acid
Scorched particle content mg	7.5 – 15	-----
Phosphate	Negative	Negative
protein %	34-37	33.4
Lactose %	49.5- 52	51
Ash %w/w	0.6 – 8.6	7.4
Fat %	0.6 – 1.25	----
Moisture %	3.0 – 4.0	4.0
Coliform mg	< 10	-----
E. coli	Negative	Negative
Salmonella	Negative	Negative
Listeria	Negative	-----
Coagulase	Positive	Positive
Staphylococci	Negative	Positive

Note:- Coagulase (positive) >10 / gram, Negative (< 10 gram), E. coli Negative in 1 gram, Salmonella (Negative) in 25 grams, Staphylococci positive (>10 / gram) negative < 10 gram),

3.3 Nutritional Value Analysis

In this study, extracting protein from the wastewater of a dairy plant to process a functional product increases the benefits of product value. The results show that wastewater coming from a dairy plant contains protein, lactose, and minerals. An effective way has to be found to separate the protein and lactose from the dairy wastewater. Concentrating the protein in milk powder is a good way to retain most of the proteins. This research utilized “spray drying”, which is a fast and safe way to process several dairy plant waste products. Spray drying has the advantage of being the most suitable process for stabilizing food industry wastewater from dairy plants (35, 36).

The powder products found in this study contain approximately 85% protein and essential amino acids. The resulting products have good bright color characteristics, a long shelf life, and the potential to act as a functional nutritional supplement. Wastewater from dairy plants has been a key consideration in both dairy products and industrial wastewater. Large amounts of dairy wastewater can hurt the environment and living organisms (37,38). As a result, the separation of protein in dairy wastewater would reduce pollution and produce a healthier environment. This study aims to show how to extract protein from the wastewater of a dairy plant and to discuss the nutritional value of the protein powder. Additionally, the study offers a discussion about how to utilize the protein concentrate coming from dairy plant wastewater. Considering all, the purpose of this study was to discuss the opportunities offered by the production of protein concentrates through spray drying, which could bring valuable additions to the highly valuable product, thus aiding the dairy industry in establishing a profitable waste management program.

3.4. Comparison Study

The dairy processing sector necessitates substantial water consumption, predominantly for equipment sanitization, vessel cleansing, and facility maintenance. Effluent generation in dairy facilities stems from three primary sources: manufacturing processes, utility operations, and auxiliary services. The operational water-to-milk ratio typically maintains a 10:1 proportion, with significant water allocation across various processing stages including milk processing, sanitization protocols, packaging operations, and transport vessel cleaning. In advanced protein-enriched milk powder production, the implementation of ultrafiltration and diafiltration technologies precedes the evaporation and desiccation phases.

The experimental treatment system employed in this investigation demonstrates reduced water requirements for cleaning operations. Statistical analysis of Table (3) data indicates superior treatment efficiency metrics, while maintaining cost-effectiveness in dairy wastewater processing. The study validates the efficacy of this system as an economically viable solution for industrial dairy effluent treatment. Dehydration represents a validated methodology for liquid food preservation, effectively inhibiting microbial proliferation and enzymatic degradation pathways. The transformation to powder form yields significant logistical advantages, including optimized transportation efficiency, simplified distribution protocols, and enhanced storage capabilities due to substantial volume reduction (39,40). Moreover, powdered formulations exhibit enhanced versatility in both domestic and industrial applications, facilitating broader implementation across various sectors(41,42).

The study analyzed treated wastewater samples, with each test repeated ten times to ensure accuracy. The results were compared with **Egyptian Law 48/1982**(43)**.,** which regulates wastewater discharge standards. The COD**,** BOD_5 **and** TSS **values (**Less than 20**)** fell within the acceptable range for discharge. Overall, the findings indicate that the sampled treated wastewater represents **lower pollution levels.**

Table 3: Comparison between biological treatment plant and dry system for the treatment of whey waste product

Pollutant	**Biological treatment plant**	**In this tudy**	law:48/1982 Secondary treated water
COD mg/L	622	Less than 20	80
BOD_5 mg/L	388	17	40
TSS mg/L	190	15	80
TOC mg/L	60	0	----

Based on the treatment process used in this study, the cost analysis of the dry system is effective and offers a lower cost than activated sludge as shown in Table (4). The cost analysis shows that the specific energy consumption of the dry milk product and treatment process is 5.5 kWh/m^3 and water consumption is 0.045 m3. The cost needed for the production of 0.14 kg of dry milk powder as a byproduct that is used in animal feed is lower in this case. This is considered a higher effective value than activated sludge plants (where energy consumption is 5.5 kWh/m^3 and water consumption is 0.045 m^3). The cost was calculated according to the following: the cost for 1 kg of dry milk powder = the cost of electricity consumed + water consumption. Hence, the cost = 0.045 + 3.935= 3.980 L.E. for the production of 1kg.

Table 4: The cost study of biological treatment plant and dry system

Process	**Biological treatment plant (L.E)**	**Dry system (L.E) in this study**
Chemical consumption* (m^3)	25	0
Electricity consumption* M^3 (kWh)	5	1.25
Operation &Maintains m^3	3.5	2.5
Total Consumption m^3	33.5	3.75

Conclusion

The dairy industry produces a significant amount of dairy wastewater and sludge during production, and the disposal of solid waste constitutes a challenge. This study managed to show how to extract protein and nutritional values from liquid dairy wastewater using "spray drying" technology. Results show that all treatments did not cause adverse effects. Overall, the feasibility of using these wastewater residues to produce protein-based lactoferrin-active and hypoallergenic ingredients was successfully validated. Furthermore, the potential application of using spray-dried dairy wastewater as a bioactive food ingredient was demonstrated. It is thus possible in the future to produce other higher value-added products from protein-enriched fractions derived from wastewater residues using additional product purification technology. For instance, unreacted alkaline after neutralization can be added back into the mixture, and membrane ultrafiltration can be further applied to purify larger fractions and produce high-value peptides that are used in the preparation of active food ingredients such as marine anti-wrinkling peptides and hydroxyproline peptides. On the other hand, further research on food, skin, and hair care applications of developing products is needed due to the increasing demand for functional products. Finally, how we can best mitigate the environmental impact is also a question of concern. The amino acid hydrolysate has a high organic concentration of nutrients, representing approximately 80% of the total chemical oxygen demand. The nutrients are easy to abate and may result in environmental damage.

Conflict of interest disclosure
No conflict interest

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