







Agricultural effect and evaluation of anaerobic digestate of the OFMSW as fertilizer on winter triticale

Efecto y evaluación agrícola de digestato anaeróbico de la FORSU como fertilizante en tritical invernal

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ABSTRACT. Anaerobic Digestion (AD) has proven to be an economically viable technology to treat the organic fraction of the municipal solid wastes (OFMSW). Research regarding the management and application of AD byproducts in Mexico, is required to encourage the private sector and local administrations to invest in this technology. In this study, fodder winter triticale (*x triticosecale Wittm. ex A. Camus* var. Río Nazas) was grown to demonstrate the fertilizer effect and to evaluate the raw digestate generated from the AD under thermophilic conditions of locally sampled OFMSW, using a batch scale digester. The digestate produced was incorporated into a 6 m² plot according to the recommender fertilization for this crop. At the same time, a crop treated with inorganic fertilizer (IF) and a control were grown, each on a plot of same dimensions, for comparison purposes. The fertilizer effect was measured in crop yield (Mg ha⁻¹), seed efficiency (kg_{Triticale} kg_{Seed}⁻¹) and bromatological value. Overall, the digestate treatment had better results in crop yield (12.54 Mg ha⁻¹) and seed efficiency (83.61 kg_{Triticale} kg_{Seed}⁻¹) than those achieved with the IF (11.16 Mg ha⁻¹ and 74.43 kg_{Triticale} kg_{Seed}⁻¹, respectively) and the control. Whereas the bromatological value, a statistical analysis revealed that, regardless of the treatment, the mineral uptake, protein, sugar and fiber content were not significantly different (p > 0.05). Raw digestate generated from AD of the OFMSW under thermophilic conditions, is a valuable by product, rich enough to fully substitute IF for growing triticale Río Nazas.

Key words: Anaerobic digestion, raw digestate, organic fertilizer, nutrient recycling, municipal waste.

RESUMEN. La digestión anaeróbica (DA) ha probado ser una tecnología económicamente viable para el tratamiento de la fracción orgánica de los residuos sólidos urbanos (FORSU). Investigación sobre el manejo y aplicación de subproductos de la DA en México, es necesaria para alentar al sector privado y administraciones locales, a invertir en dicha tecnología. En este estudio, se cultivó tritical forrajero de invierno (*x triticosecale Wittm. ex A. Camus* var. Río Nazas) para demostrar efecto fertilizante y evaluar el digestato crudo generado por la DA termófila de la FORSU muestreada localmente, utilizando un digestor a escala piloto. El digestato producido se aplicó de acuerdo con la fertilización recomendada para este cultivo, en una parcela de 6 m². A la par, se realizó un cultivo tratado con fertilizante inorgánico (FI) y otro como control de mismas dimensiones, con fines comparativos. El efecto fertilizante se midió en producción de cosecha (Mg ha⁻¹), eficiencia de semilla (kg_{Triticale} kg_{Seed}⁻¹) y valor bromatológico. El tratamiento con digestato obtuvo mejores resultados de producción de cosecha (12.54 Mg ha⁻¹) y eficiencia de semilla (83.61 kg_{Triticale} kg_{Seed}⁻¹) con respecto al tratamiento de FI (11.16 Mg ha⁻¹ y 74.43 kg_{Triticale} kg_{Seed}⁻¹, respectivamente) y el control. En valores bromatológicos, el análisis estadístico reveló que, sin importar el tratamiento, el contenido de minerales, proteína, azúcar y fibra no varió significativamente (p > 0.05). El digestato crudo generado por la DA termófila de la FORSU es un subproducto valioso, suficientemente enriquecido para substituir completamente el FI en cultivos de tritical Río Nazas.

Palabras clave: Digestión anaerobia, digestato crudo, fertilizante orgánico, reciclado de nutrientes, residuos municipales.

INTRODUCTION

Inadequate management of the OFMSW can derive in health and environmental consequences (Hosseini *et al.* 2019, Iocoli *et al.* 2019). Anaerobic digestion (AD) has proven to be a cost-effective organic waste treatment technology (Goux *et al.* 2016). Apart from the biogas, there is a second byproduct obtained through AD called digestate, which is the remaining sludge composed of a combination of partially degraded organic matter, biomass and inorganic compounds, most of which, contain readily available nitrogen, phosphorus and potassium (NPK), compounds that plants can absorb as nutrients (Gerardi 2003). These characteristics have permitted digestate to be effectively used as a soil amendment and as partial to full substitute for commercial fertilizers (Ehman *et al.* 2018, Akhbar *et al.* 2021, Seruga and Krzywonos 2021).

Mexico has over 24 million ha being used for agriculture. In the year 2017, only 4.9 million tons of the more than 23 million tons of the total inorganic fertilizer required, were locally produced (CEDRSSA 2018). The fertilizer production gap and its transportation carbon footprint (Howarth 2008, De Boer *et al.* 2019) might be reduced while managing organic waste through AD if digestate is used as a crop fertilizer. There are few anaerobic digesters installed in Mexico, most of which are fed with cow and pork dung, industrial organic wastes, or a mix of them (Gutiérrez 2018). The usage of digestate as soil amendment is already a common practice of the few digester's owners and it is usually applied on livestock's feed crops such as wheat, sorghum, oat, or triticale (Ramírez *et al.* 2019). The latter (*x tritico-secale Wittm. ex A. Camus*) is a hybrid of wheat and rye, with relatively high crude protein content (Wioletta *et al.* 2020). It is known to be drought resistant and capable to adapt to a broad range of temperatures, pH levels, salinity concentrations in soil and relatively high pest and disease resistant (Montemayor *et al.* 2017, GRDC 2018, Glamočlija *et al.* 2018). Apart from that, little is known in Mexico about the AD of OFMSW and the fertilizer value of its raw or untreated digestate (Campuzano and González 2016,

Tsydenova 2019).

To the best of our knowledge, no reports have been made in the use of raw digestate generated by the AD of the OFMSW under thermophilic conditions, as a N and P fertilizer substitute for triticale crops in México. The aim of this study was to demonstrate the fertilizer effect and evaluate the raw digestate generated from the AD of locally sampled OFMSW under thermophilic conditions on winter triticale (*x tritico-secale Wittm. ex A. Camus* var "Rio Nazas").

MATERIALS AND METHODS

Digestion process

The digestate used in this work, was collected from a 120 L 316 grade stainless steel digester built for this research, to treat locally sampled OFMSW under thermophilic and dry conditions (TS > 15%). Three single-stage, batch mode AD of the OFMSW where consecutively performed on a 6-month period. The digester working volume used was 50% of total capacity, i.e., 60 L. Industrial grade N₂ gas was used to purge air out from the system. The AD batch experiments were performed under thermophilic conditions at 55 °C ± 2. The digester content was stirred 2 min day⁻¹ to promote bacterial growth and ensure the best performance while maintaining energy consumption as low as possible (Singh *et al.* 2020). The biogas production was measured every day and its composition was analyzed every 2 days; the method is further described. Each AD batch lasted 50 days.

OFMSW and inoculum

The OFMSW used as feedstock was sampled from the Torreon City's Central Market on May, July and September 2019, respectively. These samples were chopped in approximately 1 cm² pieces and divided in two parts; one small portion, around 2 kg were used for physical and chemical analysis, the rest was kept at 4 °C until the AD experiments started.

The inoculum used was collected from a leachate generated by the mature compost piles located inside Bordo Poniente's Compost Plant in Mexico City and pre-treated with a 15-day incubation at 50 °C (Gállego *et al.* 2019). The percolated

leachate sample was taken on April of 2019.

Digestion set-up

For the first AD run, the feedstock was inoculated with the pre-treated leachate on a 1:1 ratio in a wet weight basis (w/w). For the two subsequent digestions, half of the digestate generated from a previous batch was used as inoculum in 1:1 ratio w/w.

Anaerobic digestion of OFMSW. Biogas generation and composition

The composition of biogas was determined using gas chromatography as described by Gállego *et al.* (2019). The biogas was measured through water column displacement method (Kawai *et al.* 2014, Martin-Ryals *et al.* 2015). The biogas and methane data were normalized (0 °C and 1 atm) and expressed as $\text{m}^3 \text{CH}_4 \text{kgVS}_{added}^{-1}$.

The digestate generated on each batch was stored in a sealed container, until enough was gathered to supply the needs of the field experiment. Residual biogas generated during digestate storage was neither reported in this work nor taken into consideration in the overall methane production.

Physical-chemical analysis

Feedstock, inoculum, digestate and soil were characterized employing standard methods. pH and electrical conductivity (EC) were measured with the electrode HANNA Instruments HI99300, while the oxidation-reduction potential was measured with an electrode HANNA Instruments H1003. Total solids (TS) were determined by drying the samples at 70 °C for 48 h. To measure volatile solids (VS), part of the dried samples was calcined at 550 °C for 2 h (Sadzawka *et al.* 2005). Klason lignin (KL) content was measured according to the method of TAPPI (2006). Total Kjeldahl Nitrogen (TKN) was analyzed using fresh samples employing a Büchi equipment (SpeedDigester K-436 and Distillation Unit K-350, BÜCHI, Switzerland) and the results were expressed as TKN% in dry weight basis.

Macroelements (Ca, Mg, K and P) and heavy metals (As, Cd, Cr, Cu, Pb, Ni, Zn and Hg) were also analyzed. To do so, the samples were dried at 70

°C for 48 h. Around 0.5 g of each sample was digested in a mixture of 5 ml of hydrochloric acid 30% Suprapur, 2 ml of nitric acid at 65% Suprapur and 5 ml of hydrogen peroxide solution $\geq 30\%$. This was maintained in constant heat, at 60 °C for 4 h. Finally, the mixture was filtered using Whatman paper 40 (Cat# 1440-150), re-suspended in 25 ml of distilled water and evaluated through atomic emission spectrometry (Optima 4300 DV, Perkin Elmer, USA). The results were expressed as percentage in dry weight basis.

The sanitary aspects consider the presence of *Salmonella* ssp. (SSP) and fecal coliforms (FC), according to the Mexican federal standard for biosolids NOM-004-226 SEMARNAT-2002. These were only analyzed in the digestate, which is considered a biosolid. The SSP and FC population was enumerated using 5-tube most probable number (MPN) technique. For presumptive test, lactose broth was used incubating at 35 °C for 24 h, while *E. Coli* broth was employed for confirmative test incubating at 44.5 °C for 24 h. The results were expressed as MPN g^{-1} . The presence of SSP was determined by resuspending 4 g in 36 ml in both tetrathionate broth and selenite cystine broth for an incubation period of 24 h at 35 °C, from these cultures, aliquots were inoculated in three different agars (XLD, SS and Bismuth sulfite) in triplicates, employing streak plate method at 35 °C for 24 h.

Field experiment

The experiment was settled inside the experimental station of the Autonomous Agrarian University Antonio Narro (UAAAN) located in the city of Torreón, in the state of Coahuila in northern Mexico (25° 33' 28.39 N, 103° 22' 18.65 O, WGS84, 1 120 masl).

A sub plot arrangement was used to demonstrate the fertilizer effect of digestate with fodder winter triticale through the comparison of the crop production rate, measured in kg of dry mater per hectare (kg ha^{-1}), the seed efficiency measured in kg of triticale in dry weight produced per kg of triticale seed ($\text{kg}_{Triticale} \text{kg}_{Seed}^{-1}$) and the bromatological value between the harvest produced in soil treated with digestate against the one produced in soil treated with inorganic fertilizer and one with neither digestate

nor inorganic fertilizer, used as a control.

A 30 m² plot was cleared and leveled off, after which it was sub divided in five adjacent 6 m² sub-plots (SP). Three sub-plots (18 m² in total), were used for a particular treatment and the other two were used as spacers, to avoid interference between treatments; SP 1 had digestate added as organic fertilizer treatment (OFT), SP 2 had no treatment and was used as spacer, SP 3 had urea mixed with ammonium-dihydrogen-phosphate added as inorganic fertilizer treatment (IFT), SP 4 was a second spacer and SP 5 was used as a control (CT).

Soil samples of the experimental field were analyzed to calculate the amount of fertilizer required to meet the recommended fertilization for this crop and to report the overall soil c The sampling for the physical and chemical characterization of the soil, was performed by randomly taking twelve-10 L samples from within 100 m² of the experimental station, at around a 0.3 m depth. The area was sub divided in equally sized quadrants i.e., 25 m². On each quadrant area, four samples were taken randomly then mixed thoroughly over a flat covered area, resulting in one compound pile. This pile was divided in quarters, two were eliminated. The leftovers were mixed again and divided in quarters for a second time, again two were extracted and the rest was stored in a sealed container and labeled as one sample. This resulted in four compound samples, which were used for analysis.

Winter fodder Triticale (*x Triticosecale Wittm. ex A. Camus*) var. Río Nazas was used for this experiment. This seed was produced in México in the state of Durango, it had a 97% purity and a minimum of 85% germination guarantee. Before shipping, the seeds were pre-treated with 1.5 L of Carbothiram and Lorsban per Mg of triticale as fungicide and pesticide, respectively. This were acquired through "Semillas Correa Mexicana S.A. de C.V." with batch code TCLRNSMC 08/19. This variety was selected due to its adaptability to the region's climate on winter, its nematode and disease resistance and its increasing usage as feedstock for the more than 1.4 million livestock (bovine, porcine, ovine and goat) registered in the Laguna county (SIAP 2020).

According to the seed supplier, the recommended fertilization for fodder winter triticale var. Río Nazas, is 160 kg ha⁻¹, 100 kg ha⁻¹ and 00 kg ha⁻¹ of N, P and K respectively. The amount of inorganic fertilizer was easily calculated for the SP3, while considering the already available nutrients in soil. On SP1 however, the digestate quantity was calculated based solely in the N ha⁻¹ recommendation, since one of the objectives of this work is to use the digestate as it is i.e., raw, with no further treatments. This implied that the amount of P was not adjusted to meet the recommended fertilization. It is also important to notice that other components such as organic matter, water, biomass and nutrients such as K, Ca, Mg, Fe and probably trace minerals, enclosed in the digestate were added. The amount of digestate and inorganic fertilizer added per SP while considering the already present nutrients in soil, is further detailed in results.

The digestate was applied on SP1 two weeks before sowing, in November 2019, by spreading it on top of the soil and mixing it within an approximate 20 cm of depth, after which, a 15 cm waterlogging irrigation sheet was added. All on the same day, to promote soil absorption and to avoid N losses via volatilization of NH₃ (Albuquerque *et al.* 2012, Riva *et al.* 2016). The inorganic fertilizer was applied one week before sowing in November 2019, using the same application method as with the digestate.

The sowing rate was 150 kg of triticale seed per ha; therefore, in November 2019, 90 g of triticale seeds were spread by hand over each 6 m² SP and tilted in shallow soil. After which, a 5 cm waterlogging irrigation sheet was applied on each. Three more auxiliary waterlogging irrigations where performed; these were on days 26, 61 and 82 after sowing (DAS). The triticale was harvested as soon as the crop reached the early soft dough stage (GRDC 2018, Gissén *et al.* 2014) these took between 117DAS to 123DAS. After harvest, there were no further seasonal repetitions of the field experiment.

Bromatological value of triticale

For crop analysis, 10 randomly selected plants from each SP were cut whole, just above ground, on

the same day of harvest; crude protein (CP) content was determined by the Kjeldahl method (6.25 factor), neutral detergent fiber (NDF) and acid detergent fiber (ADF) were analyzed using the method described by Soest *et al.* (1991). The rest of the crop production was also cut whole and packed in bundles, to set for drying to estimate crop yield and seed efficiency.

Statistical analysis

The reported data regarding physical and chemical analysis, as well as the bromatological values, were submitted to variance analysis (ANOVA) and the means were compared by Tukey test at $P < 0.05$ for significant differences.

RESULTS

ORP, VS and pH are key parameters to notice in the AD process, in this regard the OFMSW had positive ORP value of 242.78 ± 4.53 , 187.36 ± 17.01 g kg⁻¹ of VS, and an acidic pH of 5.10 ± 0.27 , in contrast the inoculum and the resulting digestate had a highly negative and significantly different ($P < 0.05$) ORP values of -413.00 ± 14.73 and -374.3 ± 45.01 relatively, which is known to indicate a highly reductant state of anaerobiosis. The VS and pH were also significantly different than the OFMSW values ($P < 0.05$) as expected after a complete AD process. The content of N, P, K, in digestate samples were of particular interest, resulting in 64.99 ± 7.43 g kg⁻¹, 14.02 ± 9.8 g kg⁻¹ and 129.13 ± 34.3 g kg⁻¹ on a dry weigh basis, respectively. Cd and Hg as well as As were not detected in neither the feedstock, inoculum nor the digestate which reflects in the absence of these toxic metals in the digester structure. Cu, Pb and Zn values in digestate were higher than what was detected on the feedstock and inoculum, nonetheless all values were below the limit of federal standard for biosolids NOM-004-226 SEMARNAT-2002. These and the rest of parameters are shown in Table 1.

Whereas the sanitary aspects of digestate, none of the agar plates showed growth of typical colonies (translucent, opaque and/or black dotted at the center) resulting in negative for SPP in 4 g of digestate. The FC population was 6 MPN g⁻¹, showing

presence of the bacteria but well below the limit value stipulated in the Mexican standard biosolids - NOM-004-SEMARNAT-2002 i.e., <1000 MPN g⁻¹ on dry weight basis.

After three 50 day-period per batch, of anaerobically digesting the OFMSW, the average Nm³ CH₄ generated per kg of added VS (kg VS_{added}) was 0.19 ± 0.04 Nm³ CH₄ kgVS_{added}⁻¹. The CH₄ content in the biogas was $66.76\% \pm 10.07\%$. The total average production of CH₄ per AD batch was 1.07 ± 0.24 Nm³.

The soil had a Clay-Loam texture, composed of 27% sand, 29% clay and 44% silt, with an alkaline pH of 8.21 ± 0.21 and paste extracted EC of 3.2 ± 0.5 . The CaCO₃% content was $17.65\% \pm 5.54$ which is classified as calcareous. Approximately 33.6 kg ha⁻¹ of the 160 kg ha⁻¹ needed for the recommended total N were already present in the soil as NO₃-N, furthermore, 61.9 kg ha⁻¹ of the 100 kg ha⁻¹ needed of total P were also available in the form P₂O₅. The rest of the macro and micronutrients already present in the soil within the experimental field are described in Table 2.

Considering what was already available in soil (Table 2), the addition of organic and inorganic fertilizers needed to meet this recommendation per SP was as follows: 32.8 kg equivalent to 54,609.49 kg ha⁻¹ of digestate were applied on SP1, corresponding to 126.33 - 46.71 - 371.97 kg ha⁻¹ of NPK respectively. For SP3, 139.6 g of urea (46-00-00) and 96.83 g of ammonium-dihydrogen-phosphate (16-54-00) equivalent to 232.69 kg ha⁻¹ and 161.13 kg ha⁻¹ respectively where applied, which added to 126.33 - 38.04 - 0 kg ha⁻¹ of NPK accordingly.

After 117 DAS the SP1 crop reached soft dough stage, whereas SP3 and SP5 took 123 DAS to reach this stage. The bromatological composition of triticale from OFT (SP1), IFT (SP3) and CT (SP5) had few significant differences ($P < 0.05$) i.e., higher content of ashes, higher content of cellulose and sugars with lower Mg uptake in CT, among others. Therefore, regardless of the treatment, the mineral uptake, protein, sugar and fiber content were not significantly different ($P > 0.05$). The bromatological values of the samples taken from each SP are described in Table 3.

After 135 DAS the crop was harvested, its yield

Table 1. Characterization of OFMSW samples used for AD and first inoculum.

Parameter	Feedstock	Inoculum	Digestate
pH	5.10 ± 0.27	8.08 ± 0.02	8.65 ± 0.55
ORP (mv)	242.78 ± 4.53	-413.00 ± 14.73	-374.3 ± 45.01
EC (mS cm ⁻¹)	8.68 ± 1.09	38.23 ± 0.31	38.45 ± 2.15
Total Solids (%)	23.63 ± 1.69	8.87 ± 0.71	6.05 ± 1.35
VS (g kg ⁻¹)	187.36 ± 17.01	38.69 ± 3.13	27.22 ± 13.75
KL (%VS)	15.27 ± 0.88	N/D	27.35 ± 0.78
TKN *g kg ⁻¹	22.76 ± 0.69	30.89 ± 3.58	64.99 ± 7.43
P* g kg ⁻¹	3.64 ± 0.54	4.47 ± 0.011	14.02 ± 9.8
K* g kg ⁻¹	19.19 ± 2.40	19.16 ± 1.24	129.13 ± 34.3
Mg* g kg ⁻¹	4.26 ± 0.25	7.55 ± 0.19	8.58 ± 0.49
Ca* g kg ⁻¹	21.21 ± 2.03	33.34 ± 0.16	24.27 ± 4.85
Cr* mg kg ⁻¹	25.01 ± 14.7	46.9 ± 28.5	21.88 ± 4.21
Cu* mg kg ⁻¹	14.80 ± 1.60	25.0 ± 6.45	53.10 ± 7.1
Pb* mg kg ⁻¹	7.60 ± 5.00	13.6 ± 1.60	19.9 ± 5.55
Ni* mg kg ⁻¹	14.00 ± 7.50	26.0 ± 13.10	13.97 ± 2.9
Zn* mg kg ⁻¹	120.7 ± 26.7	178.1 ± 4.2	223.6 ± 29.9

*Dry weight basis. ORP: Oxidation-reduction Potential. EC: Electrical Conductivity. VS: Volatile Solids. KL: Klason lignin. TKN: Total Kjeldahl Nitrogen. N/D: Non detectable. N/A: Not analyzed.

Table 2. Physical-chemical characterization of soil, taken at the experimental station of UAAAN.

Parameter	Value	Estimated kg ha ⁻¹ (0.3m depth)
pH	8.21 ± 0.21	-
EC (mS cm ⁻¹)	3.20 ± 0.50	-
Soil density (g cm ³⁻¹)	1.43 ± 0.08	4 293 387.55
CaCO ₃ (%)	17.65 ± 5.54	7 57 782.90
Total Solids (%)	96.70 ± 1.72	3 916 350.00
%VS *	8.10 ± 1.12	328 050.00
%Organic Matter *	3.55 ± 1.72	222 750.00
N * mg kg ⁻¹	7.84 ± 1.79	33.67
P * mg kg ⁻¹	14.43 ± 1.32	61.96
K * mg kg ⁻¹	1,576.13 ± 247.29	6 875.48
Ca * mg kg ⁻¹	3,605.44 ± 432.92	17 386.29
Mg * mg kg ⁻¹	283.18 ± 52.45	958.48
Na * mg kg ⁻¹	930.16 ± 361.38	4 229.88
Fe * mg kg ⁻¹	4.22 ± 0.42	18.35
Cu * mg kg ⁻¹	0.92 ± 0.59	3.99
Zn * mg kg ⁻¹	7.68 ± 1.97	30.78

* Dry weight basis. Estimations on kg ha⁻¹ assume that there are ~4 293 387 kg of soil on 1 ha within a 30 cm depth, assuming a soil density of 1.43 g cm³⁻¹ ± 0.08.

in dry weight, was 12.54 t ha⁻¹, 10.63 t ha⁻¹ and 6.39 t ha⁻¹ for SP1, SP3 and SP5 respectively. Hence, the seed efficiency (crop yield in kg_{Triticale} kg_{Seed}⁻¹) was 83.61, 74.43 and 43.62 Mg ha⁻¹ as shown in Table 4. These last results indicate that the OFT(SP1) was better at facilitating seed germination and development. Considering the crop yield, a total bromatological output can be estimated, i.e., the nutrient content of the triticale crop harvested in Mg per ha is shown in Table 5.

DISCUSSION

Most of the analyzed parameters of the OFMSW such as TS, VS, pH and TKN had no significant difference ($P < 0.05$) with the broad range of values reported in the literature (Tampio *et al.* 2016, Ahmed *et al.* 2021,). This was reflected in the CH₄ production by achieving quantities within the average reported by Ahmed *et al.* (2021) and Sailer *et al.* (2021). This allows to have some degree of certainty of the outcomes when using the OFMSW as feedstock in AD.

Table 3. Bromatological composition of triticale according to the fertilizer tested.

Treatment	OFT	IFT	CT
Ashes %	9.70 ± 1.64 ^{ab}	10.77 ± 0.56 ^a	7.66 ± 0.78 ^b
Cellulose %	27.99 ± 0.96 ^a	28.41 ± 0.87 ^{ab}	30.31 ± 0.24 ^b
Hemicellulose %	20.76 ± 0.78 ^a	20.13 ± 1.09 ^a	18.96 ± 0.83 ^a
Lignin %	1.52 ± 1.24 ^a	1.50 ± 0.20 ^a	1.01 ± 0.75 ^a
Starch %	9.66 ± 1.71 ^a	11.72 ± 2.07 ^a	8.58 ± 0.17 ^a
Protein %	11.83 ± 1.08 ^a	10.86 ± 0.78 ^a	10.38 ± 0.33 ^a
Sugars %	7.96 ± 0.25 ^{ab}	6.44 ± 0.36 ^a	9.25 ± 1.17 ^b
Ca %	0.16 ± 0.01 ^a	0.10 ± 0.01 ^b	0.10 ± 0.01 ^b
Mg %	0.09 ± 0.01 ^a	0.07 ± 0.01 ^{ab}	0.05 ± 0.01 ^b
Na %	0.19 ± 0.01 ^a	0.16 ± 0.05 ^a	0.23 ± 0.05 ^a
K %	0.98 ± 0.32 ^a	1.16 ± 0.15 ^a	0.63 ± 0.23 ^a
Cl %	0.13 ± 0.07 ^a	0.17 ± 0.04 ^a	0.16 ± 0.03 ^a
S %	0.12 ± 0.03 ^a	0.10 ± 0.01 ^a	0.08 ± 0.01 ^a

All values are expressed in dry weight basis. a,b,c. Values with different superscripts are significantly different (P < 0.05). OFT: Organic fertilizer treatment, IFT: Inorganic fertilizer treatment, CT: Control treatment.

Table 4. Winter triticale yield results after each treatment.

Treatment	Fertilizer	Comprised of	NPK added kg ^β ha ⁻¹	Sowing rate kg ha ⁻¹	Crop Yield ^{ββ} t ha ⁻¹	Seed efficiency ^{βββ} kg ^{Triticale} kg ^{Seed} ⁻¹
Control	No fertilizer	Experimental station soil	00-00-00	150	6.55	43.62
Inorganic Fertilizer	Urea 16-54-00	CH ₄ N ₂ O, (NH ₄)H ₂ PO ₄	126-38-00	150	11.16	74.43
Organic Fertilizer	Digestate	OFMSW	126-46-372	150	12.54	83.61

^β NPK added dose is based on triticale seed supplier recommendation of 160-100-00 fertilization. The addition was made considering the already available nutrient in soil (Table 2). ^{ββ} Crop yield is expressed in whole triticale plant on dry weight basis. ^{βββ} Triticale seed efficiency is expressed in kg of whole triticale plant on dry weight basis per kg of seed applied.

Table 5. Crop yield and bromatological value t ha⁻¹.

Treatment	OFT	IFT	CT
Crop Yield	12.54	10.63	6.39
Ashes	1.21 ± 0.20 ^a	1.20 ± 0.06 ^a	0.50 ± 0.05 ^b
Cellulose	3.51 ± 0.12 ^a	3.17 ± 0.97 ^b	1.98 ± 0.02 ^c
Hemicellulose	2.60 ± 0.10 ^a	2.25 ± 0.12 ^b	1.24 ± 0.05 ^c
Lignin	0.19 ± 0.15 ^a	0.17 ± 0.02 ^a	0.07 ± 0.05 ^a
Starch	1.21 ± 0.21 ^a	1.31 ± 0.23 ^a	0.56 ± 0.01 ^b
Protein	1.48 ± 0.13 ^a	1.21 ± 0.09 ^b	0.68 ± 0.02 ^c
Sugars	0.99 ± 0.03 ^a	0.72 ± 0.04 ^b	0.60 ± 0.08 ^b
Ca*	20.07 ± 1.25 ^a	11.54 ± 0.64 ^b	6.76 ± 1.00 ^c
Mg*	11.29 ± 1.25 ^a	7.44 ± 1.29 ^b	3.27 ± 0.65 ^c
Na*	23.41 ± 1.92 ^a	18.24 ± 5.73 ^a	15.27 ± 3.29 ^a
K [†]	122.91 ± 41.06 ^a	129.13 ± 17.31 ^a	41.00 ± 15.52 ^b
Cl*	15.89 ± 8.54 ^a	18.98 ± 4.47 ^a	10.47 ± 1.23 ^a
S*	15.05 ± 3.76 ^a	10.79 ± 1.71 ^{a-b}	5.02 ± 1.00 ^b

All values are reported on dry weight basis and expressed in t ha⁻¹ except for * Kg ha⁻¹. a,b,c: Values with different superscripts are significantly different (P < 0.05). Organic fertilizer treatment, IFT: Inorganic fertilizer treatment, CT: Control

Whereas the digestate generated, after comparing the N value found with, those reported by Tampio *et al.* (2016), Peng and Pivato (2019) and Akhbar *et al.* (2021), we observe that regardless of the treatment conditions, if the OFMSW is used as feedstock, its raw digestate show similar values of N most of

which will be present as NH₃ or NH₄ compounds, readily detectable via the TKN process. The same can be said for the rest of the parameter values, most of which, fall within the average of a broad range of physical and chemical characterizations of digestates generated under similar AD conditions (Peng and Pi-

vato 2019, Akhiar *et al.* 2021, Seruga and Krzywonos 2021). These includes the quantity of toxic metals except for As, which was only considered by Tampio *et al.* (2016) who reported slightly higher values as well as Zn. Surprisingly, a parameter that we couldn't find being used in any recent and similar studies, was the ORP, which we found quite useful as an indicator of the AD process while considering the pH.

Regarding the sanitary aspects of the digestate, the absence of SPP must have been related to the thermophilic digestion used for this study. It is well known that SPP can't survive for long periods (> 90min) at 55 °C, this was also noted by Alburquerque *et al.* (2012) while using and comparing mesophilic digestion with thermophilic digestion and finding SPP in the mesophilic digestate. Likewise, the low FC population can be attributed to the constant high temperature. These results imply an advantage of working with thermophilic conditions, due to a safer handling and readily applications.

Overall, these are the parameters that put digestate in the fertilizer perspective. It is important to notice that, in practical applications, the amount of water and the pH may need some pre-treatment, quite commonly being a solid-liquid separation (Akhiar *et al.* 2021). In this case, the digestate was used as such to avoid adding post-treatment costs to the process.

The soil in which the digestate was applied, confirmed what it's typical of the region: calcareous and slightly saline-alkaline soils (Cervantes *et al.* 2007, Chesworth 2008). This includes a high quantity of K and Na which is probably related to the fact that the whole region used to be a lagoon and as water evaporated, washed salts remain in the topsoil. Considering this, adding digestate with relatively high content of K and Ca raised some concerns of possible phytotoxicity. Triticale is known to withstand elevated levels of salinity (GRDC 2018, Zhu 2018) but surely, this where not the ideal conditions.

The triticale crop experiment was performed only once with no replicate between treatments. Even though a batch scale digester was built for this work, it took more than 150 days (three 50-days batches) to generate enough digestate to fertilize 6 m² given the digester capacity. This along with budget limita-

tions resulted in the possibility of one seasonal harvest. Therefore, the crop yield and seed efficiency results should be regarded as a demonstration of the fertilizer effect of digestate and it is expected to justify further research fundings in the matter, in Mexico. Nonetheless, after the crops were harvested, the results were clear, the OFT achieved the best results in crop yield and seed efficiency (Graphic evidence shown in Online Resource DASS.pdf, Figure S3). Although better results, these were not outstanding but rather average, when compared with similar studies with triticale (Ehmann *et al.* 2018, Caruso *et al.* 2018, Gissén *et al.* 2014).

Whereas the bromatological values found in the produced triticale, the percentage of protein and sugar content were within the average values reported in the literature regarding fodder triticale produced with conventional methods (GRDC 2018). In terms of mineral content, this was not the case, apparently, the triticale here produced was able to absorb more Ca, Mg, K and S than what Gissén *et al.* (2014), GRDC (2018) and Zhu (2018) reported in similar conditions. However, according to what Zhu (2018) found, this discrepancy could probably be more related to the triticale variety than to the used fertilizer and type of soil.

Further soil amendments and irrigation adjustment must be made to at least, meet the average production reported in the literature (Gissén *et al.* 2014, GRDC 2018, Caruso *et al.* 2018), along with a larger multi-seasonal research. Nevertheless, these results support the fact that a combination of digestate with inorganic fertilizer or even a full substitution of mineral fertilizers with digestate, is feasible, which means a concomitant reduction of mineral fertilizer, therefore less impact on the environment with a substantial economic impact.

CONCLUSIONS

Digestate generated from the AD of the locally sampled OFMSW under thermophilic conditions, is a valuable byproduct with a high content of N, K, and an important amount of P along with a high amount of water, biomass, organic matter and other macro and

micronutrients. It was demonstrated that digestate is rich enough to fully substitute inorganic fertilizer in fodder winter triticale (*x triticosecale Wittm. ex A. Camus* var. Río Nazas) by outperforming the crop yield and seed efficiency of the same crop in the same conditions, treated with inorganic fertilizer and one without treatment. The bromatological values were not significantly affected, regardless of the treatment. The valorization of digestate is a key component to achieve the profitability needed to justify any AD facility project. If municipalities like Torreon, source separate as little as 10% of the daily MSW generated by the population in non-biodegradable and biodegradable fractions, using the latter for AD, around US\$ 23 000.00 could be saved each month in management and disposal fees in local landfills. The biogas produced would have its known economic benefits and the digestate would represent important

savings in fertilizers for local producers which could also replenish organic matter and biomass in local crop fields and green urban areas. It is not clear if the alkaline pH of digestate may damage soils and crops on the long run. Further research with larger multi-seasonal crops must be performed, in Mexico.

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LITERATURE CITED

- Alburquerque J, de la Fuente C, Campoy M, Carrasco L, Nájera I, Baixauli C, Caravaca F, Roldán A, Cegarra J, Bernal M (2012) Agricultural use of digestate for horticultural crop production and improvement of soil properties. *European Journal of Agronomy* 43: 119-128.
- Ahmed B, Tyagi VK, Aboudi K, Naseem A, Álvarez-Gallego CJ, Fernández-Güelfo LA, Kazmi A, Romero-García LI (2021) Thermally enhanced solubilization and anaerobic digestion of organic fraction of municipal solid waste. *Chemosphere* 282: 131-136.
- Akhiar A, Guilayn F, Torrijos M, Battimelli A, Shamsuddin AH, Carrère H (2021) Correlations between the composition of liquid fraction of full-scale Digestates and process conditions. *Energies* 14: 971. DOI: 10.3390/en14040971.
- Campuzano R, González S (2016) Characteristics of the organic fraction of municipal solid waste and methane production: A review. *Waste Management* 54: 3-12.
- Caruso C, Maucieri C, Barco A, Barbera A, Borin M (2018) Effects of mycorrhizal inoculation and digestate fertilisation on triticale biomass production using fungicide-coated seeds. *Irish Journal of Agriculture and Food Research* 57: 42-51.
- CEDRSSA (2018) Fertilizantes. Centro de Estudios para el Desarrollo Rural Sustentable y la Soberanía Alimentaria. Palacio Legislativo de San Lázaro. <https://www.cedrssa.gob.mx/files/b/9/59NOTAS%20SOBRE%20FERTILIZANTES.pdf>. Data consulted: 12 October 2019.
- Cervantes MC, Franco AM, López A (2010) Diagnóstico Inicial del medio físico. In: López AL, Crispín AS (ed) Comarca lagunera: Procesos regionales en el contexto global. Universidad Autónoma de México. Mexico Mexico. pp: 99-122
- Chesworth W, Camps-Arbestain M, Macías F (2008) Calcareous soils. In: Chesworth W (ed) Encyclopedia of soil science. Encyclopedia of Earth Sciences Series. Springer, Dordrecht. pp: 78-125.

- De Boer M, Wolzak L, Slootweg J (2019) Phosphorus: Reserves, production, and applications. In: Ohtake H, Tsuneda S (eds) Phosphorus recovery and recycling. Springer, Singapore. pp: 75-100.
- Ehmann A, Thumm U, Lewandowski I (2018) Fertilizing potential of separated biogas digestates in annual and perennial biomass production systems. *Frontiers in Sustainable Food Systems 2*: Article 12. DOI: 10.3389/fsufs.2018.00012
- FAO (2009) Soil texture. Food and Agriculture Organization of the United Nations Rome, Italy. https://www.fao.org/tempref/fi/cdrom/fao_training/fao_training/general/x6706e/x6706e06.htm. Data consulted: 15 February, 2020.
- Gállego AK, Salcedo DA, López G, Nirmalkar K, Murugesan S, García-Mena J, Gutiérrez ME, Tovar LR (2019) Microbial profile of the leachate from Mexico City's bordo poniente composting plant: An inoculum to digest organic waste. *Energies 12*: 2343. DOI: 10.3390/en12122343
- Gerardi MH (2003) The microbiology of anaerobic digesters. 2nd Edition. Wiley & Sons. New Jersey, USA. 177p.
- Gissén C, Prade T, Kreuger E, Nges IA, Rosenqvist H, Svensson S, Lantz M, Mattsson JE, Börjesson P, Björnsson L (2014) Comparing energy crops for biogas production - Yields, energy input and costs in cultivation using digestate and mineral fertilisation. *Biomass and Bioenergy 64*: 199-210.
- Glamočlija N, Starčević M, Ćirić J, Šefer D, Glišić M, Baltić M, Marković R, Spasić M, Glamočlija Đ (2018) The importance of triticale in animal nutrition. *Ветринарски Журнал Републике Српске 1*: 73-94.
- Goux X, Calusinska M, Fossépré M, Benizri E, Delfosse P (2016) Start-up phase of an anaerobic full-scale farm reactor - Appearance of mesophilic anaerobic conditions and establishment of the methanogenic microbial community. *Bioresource Technology 212*: 217-226
- GRDC (2018) Grownotes - Triticale. 1st Edition. Northern GrowNote, Canberra, AU. 24p.
- Gutiérrez JP (2018) Situación actual y escenarios para el desarrollo del biogás en México hacia 2024 y 2030. Red Mexicana de Bioenergía A.C. Red Temática de Bioenergía de CONACYT. Mexico City, Mexico. 22p.
- Hosseini E, Azizi A, Baziyar A, Hafez H, Elbeshbishy E (2019) Comparison of liquid and dewatered digestate as inoculum for anaerobic digestion of organic solid wastes. *Waste Management 87*: 228-236.
- Howarth R (2008) Coastal nitrogen pollution: A review of sources and trends globally and regionally. *Harmful Algae 8*: 14-20.
- locoli G, Zabaloy M, Pasdevicelli G, Gómez M (2019) Use of biogas digestates obtained by anaerobic digestion and co-digestion as fertilizers: Characterization, soil biological activity and growth dynamic of *Lactuca sativa* L. *Science of the Total Environment 357*: 11-19.
- Kawai M, Nagao N, Tajima N, Niwa C, Matsuyama T, Toda T (2014) The effect of the labile organic fraction in food waste and the substrate/inoculum ratio on anaerobic digestion for a reliable methane yield. *Bioresource Technology 157*: 174-180.
- Martin-Ryals A, Schideman L, Li P, Wilkinson H, Wagner R (2015) Improving anaerobic digestion of a cellulosic waste via routine bioaugmentation with cellulolytic microorganisms. *Bioresource Technology 189*: 62-70.
- Montemayor JA, Segura MA, Munguía J, Woo JL (2017) Productividad del agua en el cultivo de triticale (X. Tritico-secale Wittmack) en la Comarca Lagunera de Coahuila, México. *Revista Mexicana de Ciencias Agrícolas 6*: 1533-1541-1547
- Peng W, Pivato A (2019) Sustainable management of digestate from the organic fraction of municipal solid waste and food waste under the concepts of back to earth alternatives and circular economy. *Waste and Biomass Valorization 10*: 465-481.

- Ramírez F, Espinosa T, Gallegos C, Santoyo VH (2019) Bioenergy production from nopal cladodes and dairy cow manure on a farm-scale level: Challenges for its economic feasibility in Mexico. *Renewable Energy* 142: 383-392.
- Riva C, Orzi V, Carozzi M, Acutis M, Boccasile G, Lonati S, Tambone F, D'Imporzano G, Adani F (2016) Short-term experiments in using digestate products as substitutes for mineral (N) fertilizer: Agronomic performance, odours, and ammonia emission impacts. *Science of the Total Environment* 547: 206-214.
- Sadzawka A, Carrasco MA, Grez R, Mora ML (2005) Métodos de análisis de compost. Instituto de Investigaciones Agropecuarias. Serie Actas N° 30. Santiago, Chile. 152p.
- Sailer G, Silberhorn M, Eichermüller J, Poetsch, J, Pelz, S, Oechsner H, and Müller J (2021) Influence of digester temperature on methane yield of organic fraction of municipal solid waste (OFMSW). *Applied Sciences* 11: 2907. DOI: 10.3390/app11072907.
- Seruga P and Krzywonos M (2021) Separate collected versus mechanical segregated organic fractions in terms of fertilizers suitability. *Energies* 14: 3971. DOI: 10.3390/en14133971.
- SIAP (2020) Población ganadera. Secretaria de Agricultura y Desarrollo Rural - Reporte Nacional 2019. Servicio de Información Agroalimentaria y Pesquera. Ciudad de México, México. <https://www.gob.mx/siap/documentos/poblacion-ganadera-136762>. Data consulted, 12 March, 2020
- Singh B, Szamosi Z, Siménfalvi Z (2020) Impact of mixing intensity and duration on biogas production in an anaerobic digester: A review. *Critical Reviews in Biotechnology* 40: 508-521.
- Soest PJ, Robertson JB, Lewis BA (1991) Methods for dietary fiber, neutral detergent fiber, and Nonstarch polysaccharides in relation to animal nutrition. *Journal of Dairy Science* 74: 3583-3597.
- Tampio E, Salo T, Rintala J (2016) Agronomic characteristics of five different urban waste digestates. *J of Environ Manag* 169: 293-302.
- TAPPI (2006) Test method. Acid-insoluble lignin in wood and pulp (T 222 om-02). In: Technical Association of the Pulp and Paper Industry. TAPPI test methods. Atlanta, USA. pp: 157-158. <https://babel.hathitrust.org/cgi/pt?id=mdp.39015080029203&view=1up&seq=158>. Date consulted: March 5, 2019.
- Tsydenova N, Vázquez A, Martínez A, Rodríguez S, Wilches C, Pehlken A (2019) Feasibility and barriers for anaerobic digestion in Mexico City. *Sustainability* 11: 4114. DOI: 10.3390/su11154114.
- Wioletta B, Katarzyna K, Ulyana B (2020) Nutritional value of wheat, triticale, barley and oats grains. *Acta Sci Pol Zootechnica* 19: 18-28
- Zhu F (2018) Triticale: Nutritional composition and food uses. *Food Chemistry*. 241: 468-479.