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Orange Peels: New Concept in Bioenergy [ID_171]

Les écorces d'orange : une nouvelle approche en matière de bioénergie [ID_171]

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ABSTRACT. Orange peel waste is increasingly acknowledged as an effective material for anaerobic digestion, which can generate bioenergy such as biohydrogen and biomethane. However, its use as a renewable energy source is still limited on a global level. Therefore, this study is to assess the capacity for biogas production from orange peel waste, collected during the ripening period of the Thomson variety (December–February 2024).The elemental composition and structure of the orange peels biomass were assessed through CHNS-O and SEM-EDX analyses. To evaluate the efficiency of hydrolysis and mass transfer, two particle size fractions were selected: D1 (63–250 µm) and D2 (250–500 µm). Various parameters were monitored during the anaerobic digestion experiments, including pH, total sugar concentration and biogas volume. It has been found that the orange peels contain 34.66 % carbon, 44.88 % oxygen, 0.40 % nitrogen, and 7.35 % hydrogen. The high oxygen content reveals a predominance of oxygenated organic compounds, particularly carbohydrates, which are available to anaerobic microorganisms and promote rapid biodegradation. The cumulative biogas produced is about 955.5 mL and 232 mL for D1 and D2, respectively. Thereby improving microbial accessibility, diffusion, and the stages of anaerobic digestion. Furthermore, the Geompertz modified model has been employed to follow the kinetics of biogas production.

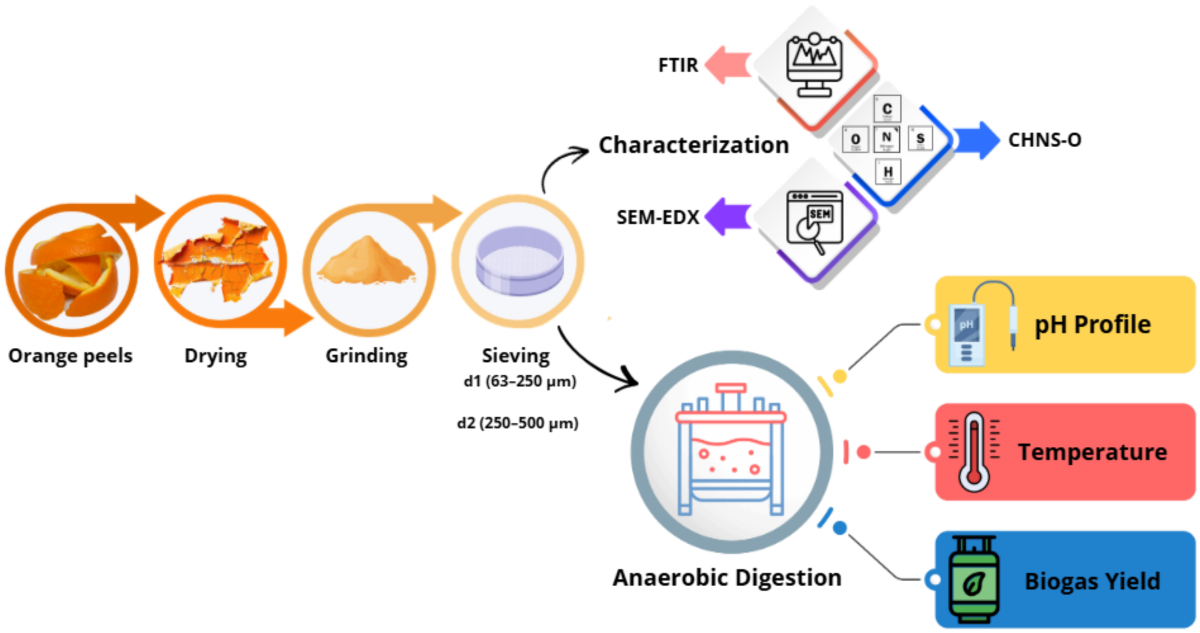
RÉSUMÉ. Les déchets d'écorces d'orange sont de plus en plus reconnus comme un matériau efficace pour la digestion anaérobie, capable de produire de la bioénergie, telle que : le biohydrogène et le biométhane. Cependant, leur utilisation en tant que source d'énergie renouvelable reste limitée à l'échelle mondiale.

Cette étude vise à évaluer le potentiel de production de biogaz à partir des déchets d'écorces d'orange collectés pendant la période de maturation de la variété Thomson (décembre-février 2024). La composition élémentaire et la structure de la biomasse des écorces d'orange ont été évaluées via les analyses CHNS-O et SEM-EDX. Afin d'évaluer l'efficacité de l'hydrolyse et du transfert de matière, deux fractions granulométriques ont été sélectionnées : D1 (63–250 µm) et D2 (250–500 µm). Divers paramètres ont été contrôlés durant les expérimentations de digestion anaérobie, notamment le pH, la concentration en sucres totaux et le volume de biogaz. Il a été constaté que les écorces d'orange contiennent 34,66 % de carbone, 44,88 % d'oxygène, 0,40 % d'azote et 7,35 % d'hydrogène. La teneur élevée en oxygène indique une prédominance de composés organiques contenant de l'oxygène, en particulier, des glucides, qui sont disponibles pour les micro-organismes anaérobies et favorisent une biodégradation rapide. La production cumulative de biogaz est d'environ 955,5 mL et 232 mL pour D1 et D2, respectivement.

KEYWORDS. Anaerobic digestion, Biogas, Orange peel residues, Characterization, modelling.

MOTS-CLÉS. Digestion anaérobie, Biogaz, Résidus d'écorces d'orange, Caractérisation, Modélisation.

GRAPHICAL ABSTRACT.



1. Introduction

The global energy crisis has highlighted the strong dependence of both developed and developing countries on fossil resources. This situation reveals the necessity to diversify ressources into alternative energy pathways [MAN 25]. In fact, organic residues have a substantial environmental impact, primarily due to their major contribution to greenhouse gas emissions [IMO 25]. So using available, abundant and low-cost biomass may offer a promising alternative solution. It can be converted into energy through thermochemical pathways, such as combustion and pyrolysis, or through biological processes involving both bacteria and methanogenic archaea, such as anaerobic digestion. This process occurs naturally in wetlands, lake sediments, rice fields, and the digestive systems of animals and insects. It relies on a consortium of microorganisms that decompose complex organic matter into simple intermediates, ultimately producing methane (CH_4) and carbon dioxide (CO_2) [MAN 25]. The final solid residue, called digestate, is rich in organic and mineral components and can be suitable for use as a fertilizer [SEA 13, PEN 18]. Hydrolysis (enzymatic degradation of macromolecules into monomers), acidogenesis (fast conversion to VFAs, H_2, and CO_2), acetogenesis (generation of acetate, H_2, and CO_2), and methanogenesis (methane from acetate and from H_2/CO_2) are the four steps of anaerobic digestion process [WHI 86, SAR 19]. The increased use of animal waste and by-products as feedstock in the biogas production process has been encouraged in line with countries' energy and environmental policies [TAG 21]. In 2024, Algeria produced around 1.3 million tonnes of fresh oranges annually, positioning it 13th in the world ranking (World Population Review, 2026). Harnessing orange peels for energy generation could serve as an effective strategy for waste conversion.

This research evaluates the potential for energy recovery through anaerobic digestion of locally grown orange peels. The elemental composition and structure of the orange peels biomass were assessed through CHNS-O and SEM-EDX analyses. To evaluate the efficiency of hydrolysis and mass transfer, two particle size fractions were selected: D1 (63–250 µm) and D2 (250–500 µm). Various parameters were monitored during the anaerobic digestion experiments, including pH, total sugar concentration, and biogas volume. Furthermore, the Geompertz modified model has been employed to follow the kinetics of biogas production.

2. Materials and methods

2.1. *Raw material*

Orange peels have been selected for biogas production due to their high content of fermentable sugars and essential nutrients, as well as their widespread availability in Algeria, which makes them suitable for a broad and diverse range of biotechnological applications. These peels were collected during the ripening period of the Thomson variety (December–February 2025), once the fresh, orange-coloured fruit with thin, smooth skin had been harvested. The biomass was subjected to drying at a temperature of 45°C for 48h and mechanical grinding prior to analysis, followed by sieving into two particle-size ranges : D1 (63–250 µm) and D2 (250–500 µm) using a BA-200N electromagnetic digital sieve shaker (CISA, Italy). This process of size fractionation aimed to assess the impact of particle size on substrate accessibility, biogas yield, and process kinetics.

2.2. *Batch digester fermentation*

The experimental setup consisted of a batch anaerobic digestion system comprising a laboratory-scale glass reactor coupled with a bell jar for biogas volume measurement. The cylindrical reactor, measuring 23 cm in height and 9 cm in diameter, was specifically designed to maintain strict anaerobic conditions. It had a total capacity of 1.2 L and a working volume of 1 L. The reactor was fitted with an airtight lid equipped with multiple ports for monitoring physicochemical parameters and a gas outlet connected to a bell jar. One port was dedicated to periodic sampling for complementary analyses, including total sugar concentration, volatile fatty acids. The reactor was filled with 50 g of orange peels in distilled water to achieve a working volume of 1L. To enhance substrate biodegradability, a mild hydrothermal pretreatment of orange peels was applied at 120°C under a pressure of 2 bars. This pretreatment promotes the disruption of complex lignocellulosic structures, thereby improving microbial accessibility, diffusion, and the stages of anaerobic digestion. The digester was operated at a constant temperature of 35±3°C under continuous agitation at 150 rpm to ensure proper mixing and homogeneity of the medium. To evaluate the performance and stability of the fermentation process, different operational parameters are monitored throughout the anaerobic digestion of orange peels, including, pH, total sugars consumption and cumulative biogas production.

2.3. *Biomass composition*

Total sugar content was quantified via visible spectrophotometry at 490 nm according to the phenol sulfuric acid method of Dubois et al. [DUB 56], using a Lambda 25 UV/Visible spectrophotometer (PerkinElmer, USA). VFA concentrations were measured by HPLC (Agilent YL9100) with a C8 column (5 µm, 25 × 0.46 cm). The mobile phase was 0.005 M H_2SO_4 at a flow rate of 0.7 mL/min. The column temperature was 30 °C, and UV detection was performed at 210 nm. Dry matter and moisture content were assessed to establish the material balance during anaerobic digestion and to provide insights into substrate degradation efficiency as well as the conversion of organic matter into energy. This involved weighing the empty crucibles, adding a known quantity of the sample and then drying them in a Memmert UM 100 drying oven at 105°C until they reached a constant weight. Subsequently, the organic matter and ash content were determined by incinerating samples at 550°C for 3 hours. This process oxidizes the organic fraction, leaving behind the mineral residue. Crucibles were weighed before and after incineration, and the results were derived from these measurements. This procedure provides essential information regarding the quantity of organic matter available for biogas production and the efficiency of the digestion process.

2.4. *CHNS-O Analysis*

An elemental analysis was performed to determine the content of carbon (C), hydrogen (H), nitrogen (N), sulphur (S) and oxygen (O) in the orange peel biomass. This CHNS-O analysis, also known as organic elemental analysis, enables the purity and chemical composition of the substrate to be assessed based on sample combustion. The elemental composition of the orange peels was determined using a Flash 2000 Organic Elemental Analyzer (Thermo Fisher), calibrated with a certified standard (Sulphanilamide, $C_6H_8N_2O_2S$) and operated through the Eager Xperience software.

2.5. *SEM-EDX Analysis*

To investigate potential morphological differences in the orange peels biomass, scanning electron microscopy (SEM) analysis was carried out. SEM is a powerful technique that provides high-resolution images of sample surfaces by exploiting the interactions between electrons and matter, allowing detailed observation of structural features that are invisible under conventional optical microscopes. The analysis enables the identification of surface characteristics such as porosity, fibre arrangement, and the presence of microfissures. These are critical parameters that influence microbial accessibility and enzymatic degradation during anaerobic digestion process. In this study, micrographs of orange peels was obtained using a FEI Quanta 650 (W) scanning electron microscope, ensuring precise visualization of surface morphology and structural differences between the selected biomasses. This morphological characterization provides essential insights into how substrate structure can affect the efficiency of anaerobic digestion and overall biogas yield.

3. Results and discussion

3.1. *Biomass composition*

The compositional analysis of orange peels (Table 1) revealed a low dry matter content of 25.41 % and a high moisture content of 74.59 %, indicating that this biomass is predominantly wet. Furthermore, orange peels exhibited a very high organic matter content of 99.12 %, highlighting their strong potential for biological conversion processes, including hydrolysis and fermentation. The ash content was found to be low at 0.88 %, reflecting a low mineral fraction. Thus, orange peels are highly biodegradable, organic-rich substrate, and well-suited for biotechnological valorization.

	Orange peels
Dry matter %	25.41
Moisture %	74.59
Organic matter %	99.12
Ash %	0.88

Tableau 1. *Biomass composition*

3.2. *CHNS-O Analysis*

The chemical characterization of orange peels is crucial for assessing their suitability for energy recovery applications. CHNS-O analysis (Table 2) indicated that orange peels contain 34.66 % carbon, 44.88 % oxygen, 0.40 % nitrogen, and 7.35 % hydrogen, with no detectable sulfur. The high oxygen content reveals a predominance of oxygenated organic compounds, particularly carbohydrates, which are available to anaerobic microorganisms and promote rapid biodegradation. Despite the low nitrogen content, which may limit microbial growth if used as a sole substrate, the carbon and hydrogen fractions contribute significantly to the overall energy potential. Moreover, the absence of sulfur is beneficial, as it minimizes the formation of hydrogen sulfide during anaerobic digestion. These findings suggest that orange peels represent a promising feedstock for biogas production, especially when co-digested with nitrogen-rich substrates to optimize process performance.

	Orange peels
Nitrogen (N) %	0.3957
Carbon (C) %	44.6568
Hydrogen (H) %	7.3469
Sulfur (S) %	ND
Oxygen (O) %	44.883

Tableau 2. *Elemental composition of orange peels determined by CHNS-O analysis*

3.3. *SEM-EDX Analysis*

Scanning electron microscopy (SEM) was employed to investigate the surface morphology and texture of the solid biomass samples. As can be seen in Figure 1, the SEM images reveal a granular structure with cavities and surface irregularities. This indicates a high specific surface area and notable porosity, which can enhance microbial accessibility during biochemical processes. Energy-dispersive X-ray (EDX) analysis showed that orange peels contain the following elements (w/w) : Mg (0.3 %), K (3.0 %), Al (0.5 %), Ni (0.2 %), Si (0.1 %), and P (0.3 %) (Table 3). This biomass highlight a distinct mineral profile, with relatively high potassium content and low levels of other elements. Ideally, it would be used for co-digestion with a Ni-poor substrate or for catalytic gasification, in which case Ni would play an active role. However, an efficient dust removal system would need to be in place.

	Orange peels
Component	0.3
Mg	3.0
K	0.5
Al	0.2
Ni	0.1
Si	0.3
P	0.3

Tableau 3. *Elemental composition of orange peels determined by SEM-EDX*

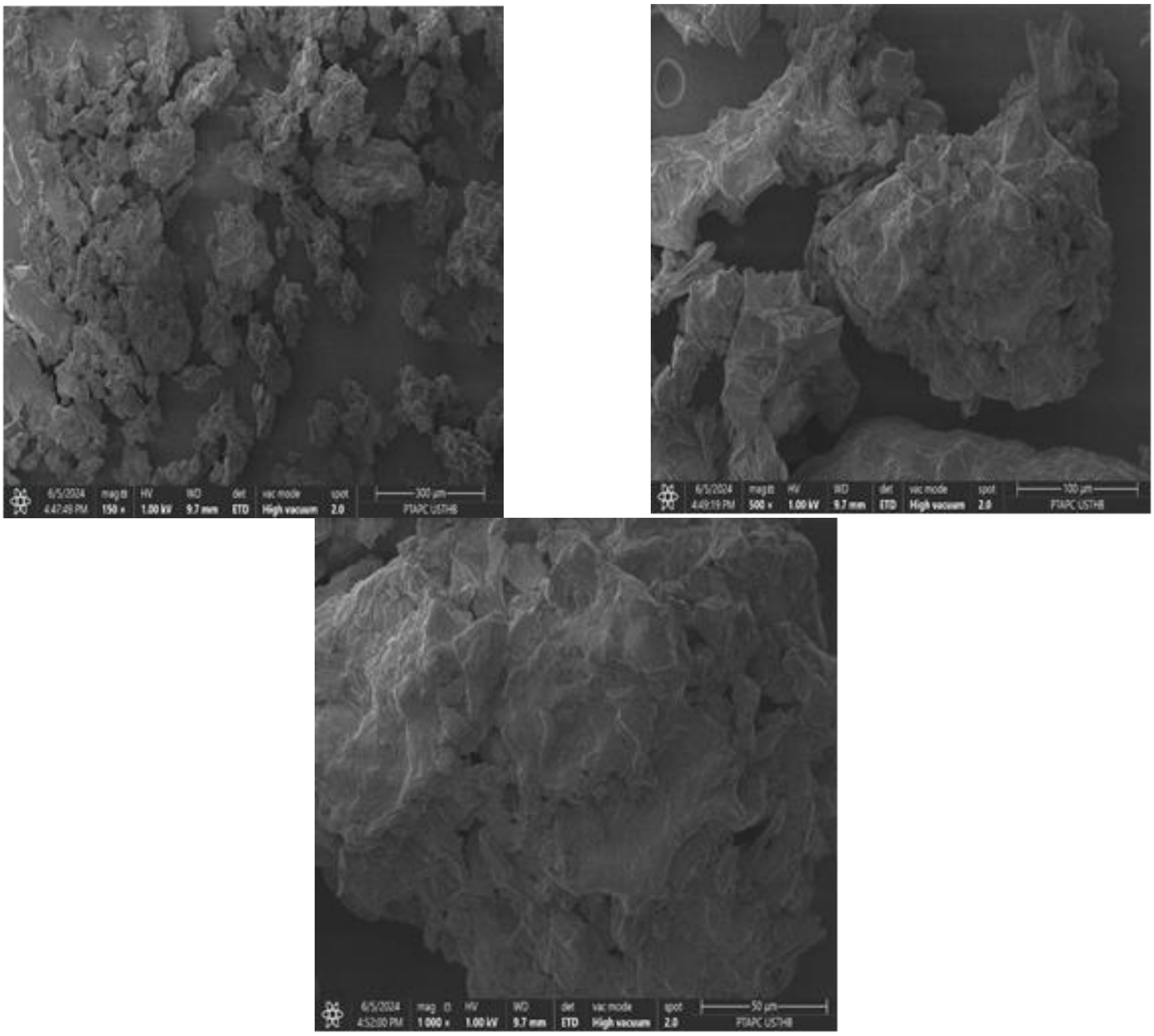


Figure 1. *SEM micrographs of orange peels*

3.4. *SEM-EDX Analysis*

The orange peels sample was analyzed via PerkinElmer Spectrum GX1 spectrometer (PerkinElmer, Inc., Waltham, MA, USA), equipped with an ATR accessory consisting of a ZnSe crystal (FTIR-ATR) (Pike Tech-nologies Miracle Universal ATR), which was used to analyse solid samples in reflectance mode.

The average spectra of orange peel sample is given in Figure 2. The discriminating elements between the two groups are mainly due to lignin, cellulose and hemicellulose, which are different in chemical composition. The ATR spectrum of the biomass feedstock reveals the presence of key functional groups associated with lignocellulosic materials. The broad absorption band observed at approximately 3267 cm^{-1} is attributed to O–H stretching vibrations, indicating the presence of hydroxyl groups associated with cellulose, hemicellulose, and bound water [MIC 23]. The peak at 2920 cm^{-1} corresponds to aliphatic C–H stretching vibrations, reflecting the presence of organic constituents such as lignin and lipids [ZAP 09]. The band around 1735 cm^{-1} is assigned to C=O stretching of carbonyl groups [DUA 17]. The absorption at 1600 cm^{-1} is related to aromatic C=C vibrations, confirming the presence of lignin structures. Furthermore, the bands at 1410 cm^{-1} [DUR 22] and 1237 cm^{-1} [ROH 15] are associated with C–H deformation and C–O stretching vibrations, respectively. A strong absorption peak at 1008 cm^{-1}, corresponding to C–O–C and C–O stretching, indicates a high content of polysaccharides, particularly cellulose [NAN 19]. Overall, the spectrum confirms the lignocellulosic composition of orange peel, highlighting the abundance of cellulose, hemicellulose, and lignin, which makes this biomass a promising substrate for biogas production following appropriate pretreatment.

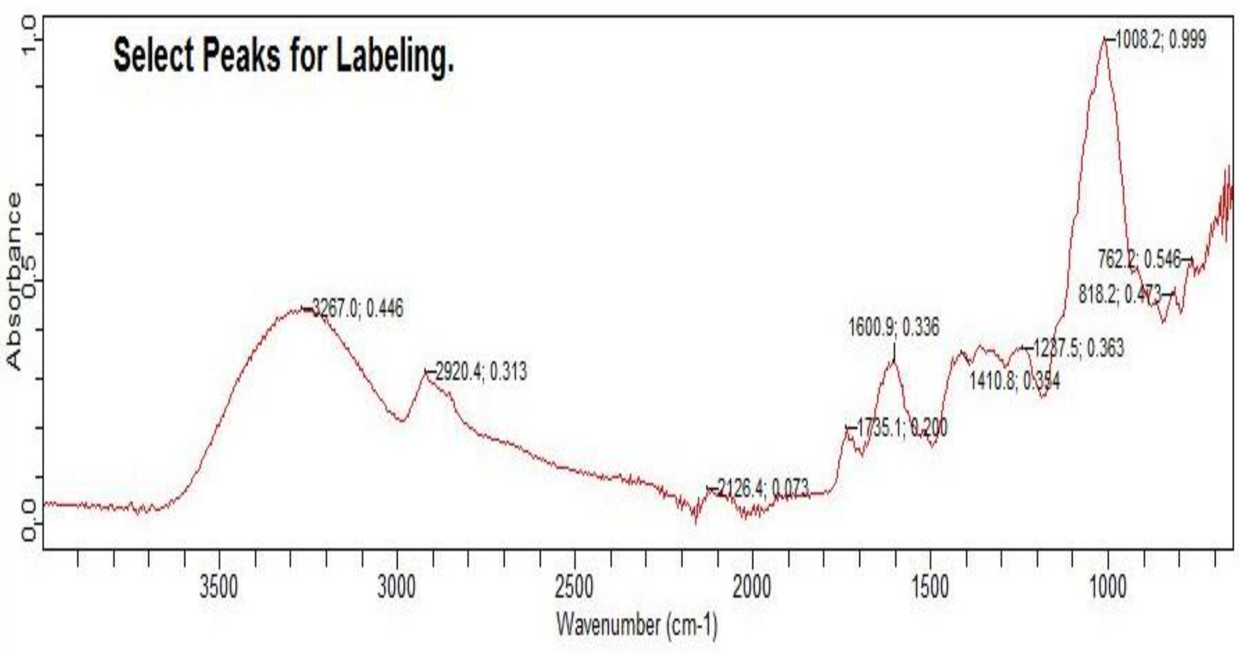


Figure 2. *ATR spectra of the orange peel biomass*

3.5. *pH profile throughout the experiment*

The monitoring of pH evolution throughout the anaerobic digestion process of orange peel substrate provides a direct indication of the system's biochemical transitions and overall stability. To assess the effect of substrate granulometry on early-stage hydrolysis and subsequent acidogenesis, two particle-size fractions, D1 (63–250 µm) and D2 (250–500 µm) were investigated. As illustrated in Figure 3, the orange peels biomass exhibited pronounced acidification during the initial phase of the process. It can be seen that the pH decreased from 7 to 3.37 and 7 to 3.71 for D2 and D1, respectively. This rapid decline indicates a significant level of hydrolytic and acidogenic activity, during which complex organic matter is degraded into soluble compounds and subsequently converted into volatile fatty acids (VFAs). A decline in pH was observed after 48 hours of fermentation, likely due to the buildup of volatile fatty acids (VFAs) [CHE 08]. This accumulation, along with the limited buffering capacity during the early stages, may inhibit effect on microbial growth and metabolic activity. Thereby limiting biogas generation, indicating a potential imbalance between the acidogenic and methanogenic phases. Nevertheless, despite the increased acidity of the medium, biogas production did not cease entirely, indicating that the microbial community retained partial functional activity under these conditions. The overall pattern of pH evolution was similar for both particle-size classes, indicating that within the tested range (63–500 µm), granulometry did not significantly affect the acidogenic phase. After this initial acidification, a progressive rise in pH was recorded.

This slight increase corresponds to the onset of acetogenic and methanogenic pathways, where VFAs and other reduced intermediates are consumed and transformed into methane and carbon dioxide. In the final phase, pH stabilization was observed across all conditions, reflecting the establishment of a balanced microbial consortium and the transition toward a steady-state digestion regime, where acid production and consumption occur at comparable rates. Some researchers [LIN 15] investigated the effect of target pH values (5.5, 6.0, 7.0, and 7.5) on the anaerobic digestion of maize silage. Their results showed that increasing the pH from 5.5 to 7.5 significantly improved substrate degradation and methane production. Specifically, the acetic acid equivalent in the hydrolysate decreased by 88.1%, while the chemical oxygen demand (COD) concentration decreased by 18.3%. In parallel, the overall system exhibited a 58% increase in specific methane yield. The effect of pH (5–9) on the digestion of food waste in batch reactors under mesophilic conditions was investigated. The results showed that the highest biogas yield and substrate degradation occurred at pH 7, with a cumulative biogas production of 5655 mL over 30 days [JAY 14].

Overall, pH directly affects microbial growth, metabolic activity, and consequently the degradation of organic matter and biogas production. When pH is maintained near neutrality, the medium remains stable and conducive to the development of microorganisms involved in anaerobic digestion, particularly methanogenic archaea, whose optimal activity occurs around pH 7 [HUM 25].

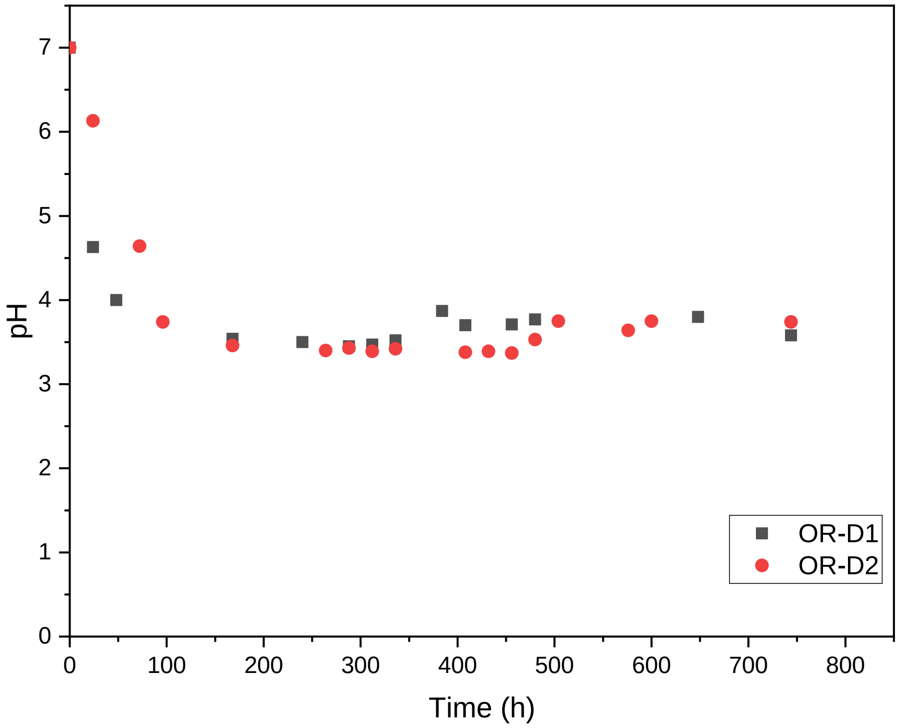


Figure 3. *Daily pH change during biogas production from orange peels under mesophilic temperature*

3.6. *Time-course of total sugars concentration*

The Sugars represent a primary source of carbon and energy in anaerobic digestion processes. The sugars released during the hydrolysis step, they are readily assimilated by microbial consortia in the early stages of fermentation, where they are converted into intermediate compounds such as organic acids, and subsequently into methane and carbon dioxide. Therefore, total sugar evolution mirrors metabolic activity and operating conditions. Accumulation of recalcitrant long-chain VFAs from fermentation can disturb process balance and indirectly affect sugar consumption. Figure 4 depicts daily total sugars change during biogas production from orange peels under mesophilic temperature. This figure shows total sugar consumption by bacteria for two particle size classes. The initial total sugar concentration is higher for class D2 (90 g/L) than for class D1 (78 g/L). The rapid decline during the first 150 hours indicates the fast uptake of readily biodegradable soluble sugars. Between 150 and 400 hours, the biomass of class D2 exhibits marked fluctuations between 59.27 g/L and 33.76 g/L, with a temporary accumulation around 65 g/L, which suggests gradual sugar release through the enzymatic hydrolysis of complex substrates. Meanwhile, the biomass of class D1 remains relatively stable at around 45 g/L. A further decrease in sugar concentration is observed for OR-D2 between 400 and 600 hours, after which both fractions stabilize at approximately 45–50 g/L for OR-D2 and 40–43 g/L for OR-D1. This suggests that easily accessible sugars have been depleted.

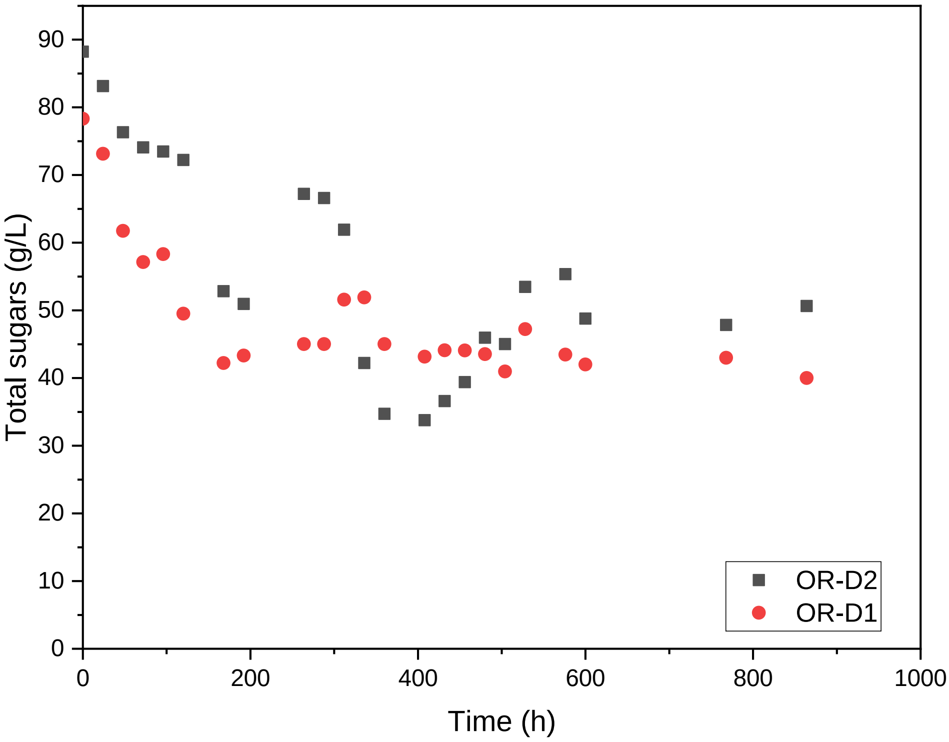


Figure 4. *Daily total sugars change during biogas production from orange peels under mesophilic temperature*

3.7. *Volatile fatty acids*

The concentrations of volatile fatty acids (VFAs) exhibit a moderate range of variation, with values spanning roughly from 150 to 700 mg/L for both orange peel granulometries Figure 5. Lower concentrations around 150 and 200 mg/L, suggest relatively stable system conditions, where VFA production and consumption are balanced. This phenomenon is probably due to bacterial metabolism, which stimulates the rate of biogas production at the beginning of anaerobic digestion [MEN 15]. Values approaching 600 to 700 mg/L suggest periods of heightened fermentative activity and potential transient acid accumulation. This may be due to a direct relationship between biomass composition, biogas production rate and fatty volatile acid accumulation [GEM 15]. Despite these fluctuations, the overall distribution remains fairly consistent, without extreme spikes, suggesting that the digester maintains a certain level of stability.

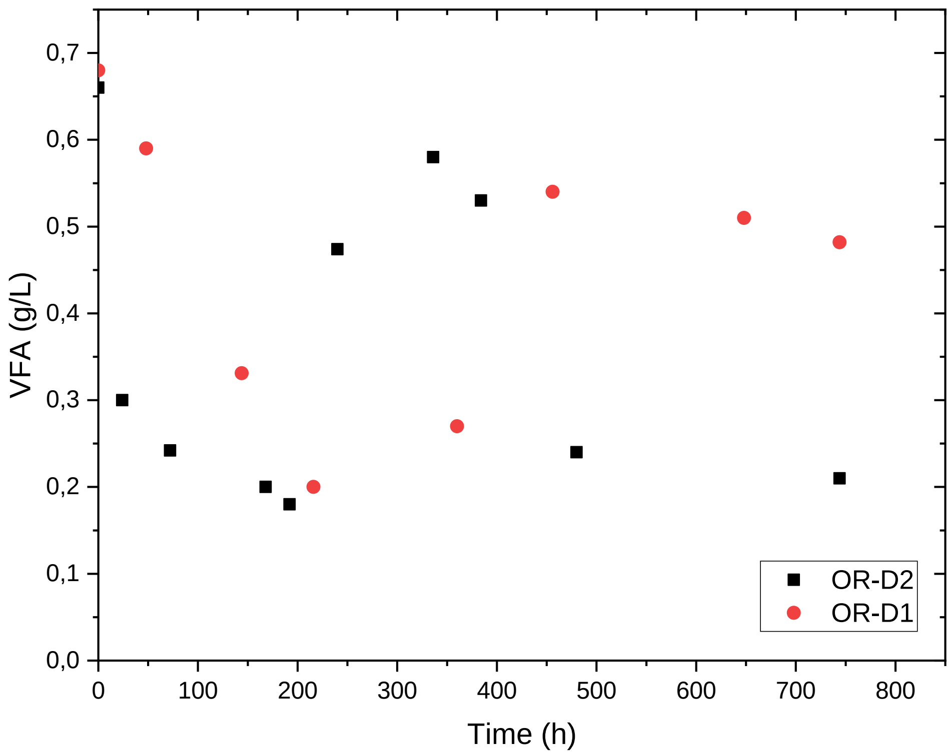


Figure 5. *VFA from orange peels under mesophilic temperature*

3.8. *Modelling cumulative biogas production*

Biogas production from orange peels was evaluated for two particle-size fractions, D1 (63–250 µm) and D2 (250–500 µm). The analysis of figure 6 indicate that the highest biogas production was obtained from OR of class D2, reaching a volume of 955.5 NmL after 24 days of orange peels anaerobic digestion. These results suggest that the particle size D2 (250–500 µm) enhances biogas production compared to D1, likely stems from better substrate accessibility to microorganisms, which promotes hydrolysis and acidogenesis- key steps in anaerobic digestion process. However, despite this high biogas yield, orange peels are known to contain inhibitory compounds, such as : essential oils, alkaloids, and phenolic compounds, including flavonoids and phenolic acids [DEL 19, NGU 12]. These substances may exert toxic effects on methanogenic microorganisms, potentially disrupting their metabolic activity and limiting the overall efficiency of biogas production. Additionally, the accumulation of volatile fatty acids (VFAs) during OR digestion may lead to a decrease in pH, which can further inhibit methanogenic activity. Nevertheless, the observed biogas production suggests that the microbial community was able to partially adapt to these inhibitory conditions.

The maximum biogas production rate is achieved with granulometry D2, which has a value of 0.0187 L biogas/g VS·h this is higher than the 0.010 L biogas/g VS·h observed for granulometry D1. This discrepancy can be attributed to the increased biogas production associated with the granulometry of orange peels in class D2. The modelled lag phase (λ) is significantly shorter for granulometry D2, recorded at 91.7 hours. This suggests that biogas production commences shortly after a certain period of digestion. In contrast, this parameter is considerably longer for class D1, at 169.7 hours. The modified Gompertz model also fits well with the experimental data for biogas production from OR biomass for both granulometry classes D2 and D1, where the R^2 is around 0.98 for D2 and 0.94 for D1.

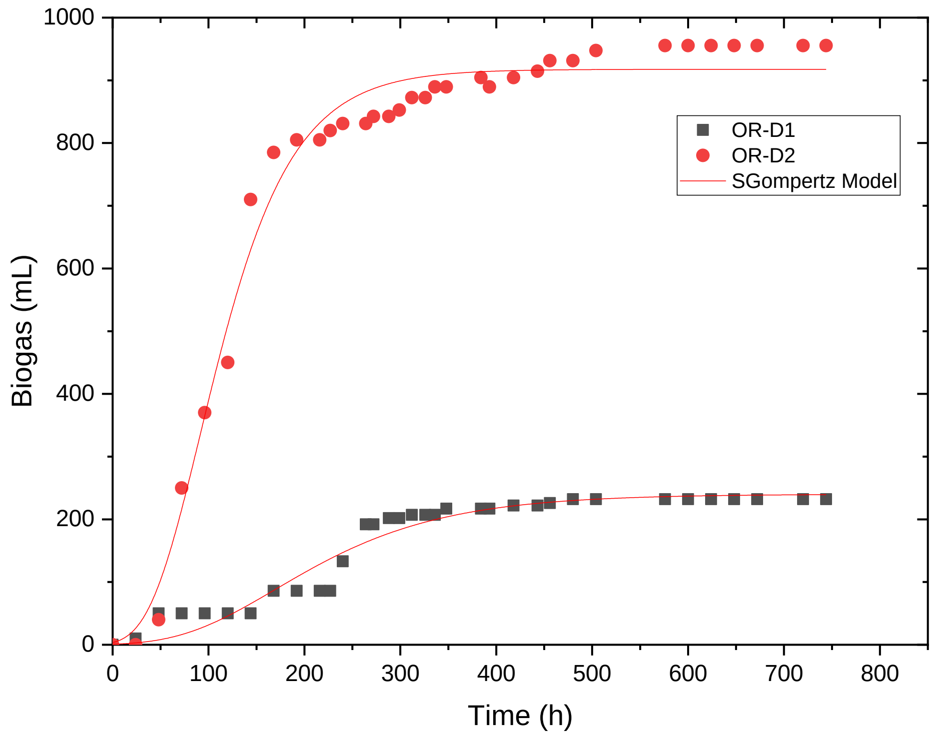


Figure 6. *Biogas production from orange peels biomass under mesophilic temperature.*

4. Results and discussion

To address both environmental and energy challenges, and based on combined theoretical and experimental investigations, the valorization of orange peels biomass for energy production appears to be a viable and sustainable option. The orange peels contain 34.66 % carbon, 44.88 % oxygen, 0.40 % nitrogen, and 7.35 % hydrogen. The high oxygen content reveals a predominance of oxygenated organic compounds, particularly carbohydrates, which are available to anaerobic microorganisms and promote rapid biodegradation. The results demonstrated that optimizing the anaerobic digestion process requires careful control of key operational parameters, including pH, total sugar content, volatile fatty acid. The highest biogas production was obtained from OR of class D2, reaching a volume of 955.5 NmL after 24 days of orange peels anaerobic digestion. In the future, the use of co-digestion involving animal manure or microalgae could improve production yields.

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