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Anaerobic digestion of recycled paper crumb and effects of digestate on concrete performance

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ABSTRACT

Paper crumb (PC) is a type of paper sludge residue from the wastepaper recycling industry. It is a by-product from the various fiber purification stages that is particularly composed of short cellulose fibers, lignin, organic compounds and inorganic filler residues. Despite representing a reject material for the paper recycling sector, this feedstock can be turned into a bioresource to enable cross-sector industrial symbiosis in the form of a more sustainable concrete, hence an opportunity for novel Net Zero supply chains. This study sought to valorise the PC by the sequential anaerobic digestion to produce methane (CH_4) from the organic compounds, followed by utilization of the digestate as a water replacement in concrete. The 21-day digestion of PC yielded 163 ml CH_4 per gram volatile solids and the resulting digestate improved concrete compressive strength up to 50% water replacement grade, meeting the requirements for structural grade (C32/40) applications with substitution grades up to 50% and 25%, with and without the addition of plasticiser respectively. In a minor capacity, the digestate reduced workability of the concrete mix, however we demonstrate this issue can be resolved by the addition of plasticiser or increased water to cement ratios. The admixture addition is important to facilitate pumpability on site and ensure satisfactory compaction. This study highlights the potential of anaerobic digestate as a concrete supplement (additive), which would improve the sustainability of both the construction and the paper sector.

1. Introduction

The use of recycled wastepaper for paper production has several environmental advantages over virgin paper, such as reduced greenhouse gas emissions, lower water consumption and reduced deforestation [1]. Recycled paper pulp accounts for over half of global paper production with more 250 million tons produced in 2018 [2]. The recycling process requires washing and pulping to remove contaminants such as inks, glue and other organic compounds such as food. However, the decontamination process also removes short paper fibers that is commonly mixed with the contaminants in various waste streams such as deinking sludge or paper crumb. The composition of the wastepaper crumb varies significantly depending on the starting wastepaper (e.g. cardboard, hygiene tissue, graphic, etc.), but accounts for the 1–4 %wt. of the feedstock [3]. [4] estimated in 2005 that over 7 million tons of waste fibers was produced, with over 500,000 tons produced in the UK alone. In recent years the landfilling of organic materials has been restricted in several countries including the UK, Germany and the Netherlands which has led to a growing interest in the valorisation of the paper crumb via alternative methods.

Most commonly, PC is dewatered and incinerated with a high associated carbon footprint [5]. The growing trend to decarbonize foundation industries will require the minimization of incineration. PC is primarily composed of the cellulose, lignin, inorganic paper fillers (primarily calcium carbonate) and organic waste compounds that could be valorised individually. Enzymatic hydrolysis has been found to successfully reduce the cellulose by up to 60% with no pH adjustment required or inhibition caused by contamination [6]. While biochar derived from deinked paper sludge has shown promising results for soil remediation from heavy metals (due to the porous carbonized cellulose-lignin structure) [7], recycled paper fibers from various sources have shown potential application in anaerobic digestion (AD)

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systems to produce methane that can be used as fuel for direct replacement in incineration [8]. The biogas produced from the AD of PC would offer a significant value addition to the paper recycling industry due to the inherent energy demands of the process, although the yields vary significantly between different initial paper feedstocks (49–167 ml CH4/gVS) that are below the calorific value of direct incineration [3,9]. The pretretment of PC with sonication, mechanical pulping and chemical pulping have been found to further increase biogas yields by breaking down the cellulose structures [10]. The AD of virgin paper fibers has shown negligible methane yields [11] indicating that the AD processes of PC only degrade the organic contaminants leaving the unreacted the cellulose, lignin and paper fillers in the post-digestion fraction, also known as digestate. Similar to the raw PC, disposal of digestate has become more troublesome in recent years due to restrictions for land applications, related to issues associated with water eutrophication and land greenhouse emissions [12]. However, the digestate from the PC could have many interesting properties relating to the cellulose, lignin and the calcium carbonate fractions, more specifically for concrete.

Recently the growing interest in low-carbon concrete has involved the use of wastewater as a water replacement for the encapsulation of carbon as a form of carbon sequestration [13]. Previous studies have shown that wastewater with solids content <10 wt% can increase the compressive strength of concrete at the expense of slower curing time [14]. The addition of wastewater changes the microstructure of the concrete by enhancing packing and reducing pore volume [15]. Similar packing effects have been found with the addition of cellulose and lignin to concrete that will be present from the incomplete PC anaerobic treatment [16]. The application of residual digestate from the AD of PC as a water replacement in concrete is very novel, with only a very limited number of studies ([17,18]); in the literature reporting on the composition of digestate and linking digestate's intrinsic properties to key concrete performance parameters (e.g. compressive strength). In addition, it should be emphasised that clean water is mostly used by contractors in the built environment at commercial scale; the use of digestate will therefore reduce potable water consumption in concrete mixing, while acting as a form carbon sequestration to partially mitigate the significant carbon implications of cement manufacture with the potential to improve mechanical strengths. The use of anaerobic digestate as water replacement in concrete is still vastly unexplored, therefore this study investigates both the AD of recycled PC to assess its methane potential, and the subsequent biochemical characterisation and use of the digestate for concrete production to evaluate an integrated waste management process between different foundation industries.

2. Methods

2.1. Materials and characterization methods

PC was acquired from a UK paper recycling company (SAICA Paper Ltd.) and immediately dried for storage in airtight containers to prevent degradation. AD inoculum was collected from a local brewery in Greater Manchester, as the microbial seed on the day of inoculations, with full compositional analysis shown in Supplementary Table 1S.

The total solids (TS) and volatiles solids (VS) were evaluated by drying overnight at 105 °C and subsequent ashing at 575 °C. The CHNSO elemental characterization was conducted in triplicate using an Elemental Vario MacroCube with O% calculated by difference, as shown in Equation (1). Minor element composition of solids was extracted using nitric acid under microwave heating and cations were analysed on a Thermo iCAP 6300 Duo ICP-OES. Water-soluble anions were determined using an ion-chromatography system equipped with a conductivity detector (ICS5000, Thermo Scientific). Cellulose content was estimated according to the international protocol NREL/TP-510-42618 [19].

$$Oxygen\ (wt.\%) = 100 - C\ (wt.\%) - H(wt.\%) - N(wt.\%) - S(wt.\%) - Ash\ (wt.\%) \quad \text{(Eq. 1)}$$

2.2. Anaerobic digestion

The biomethane potential (BMP) of PC was conducted using 500 ml sealed glass Erlenmeyer flasks under mesophilic conditions at 32 °C submerged in a water bath. The glass flasks were connected to individual biogas bags with the volume and composition measured daily for 21 days, when it was interrupted due to cumulative biogas volume production <1 %vol. The CH_4 concentration in the biogas was determined using a calibrated Geotech Biogas 5000 Plus. The biogas volumes were then converted to standard gas conditions of 0 °C and 1 atm. The experimental data was fitted to a modified Gompertz equation by [20] (shown in Eq. (2)), using non-linear regression analysis in Matlab© (2016a) where, $V_{CH4}(t)$, is the predicted cumulative methane production (mL/g VS) at any time t (day), A_{max} is the measured cumulative methane yield (mL/g VS), $R_{max}$is the maximum methane production rate (mL/g VS·d), e is the mathematical constant 2.718282, and λ is the lag phase delay (day).

$$V_{CH4}(t) = A_{max} \exp\left[- \exp\left(\frac{R_{max} * e}{A_{max}} (\lambda - t) + 1 \right) \right] \quad \text{(Eq. 2)}$$

The PC was co-digested with inoculum from a brewery and the experiments were conducted in accordance with [21]; using an inoculum: substrate ratio of 4:1. The control inoculum reactors, whose approximate microbial and proximate/elemental composition has been reported in other works [22,23], were conducted in triplicate and the PC digestion was replicated in 10 vessels to produce sufficient digestate for concrete manufacture. After the 21-day trial period the digestate samples were stored in a fridge (4 °C) overnight to limit further microbial activity and used for concrete manufacture the following day.

A biodegradability index in percentage was used to estimate the digestion efficiency via biochemical methane potential (BMP) assays. The biodegradability index has been calculated as conducted in another study [24], as the ratio of the actual methane yield to the stoichiometric methane yield, using the well-known Buswell equation [25].

2.3. Concrete mix preparation and testing

The digestate obtained as described in section 2.2 was used 'as is' as water substitute to prepare the concrete mixes, using 3 replicates per condition set (n = 5). Details of the concrete mixes are shown in Table 1, the cement used was type CEM 1 52.5N conforming to BS EN 197-1: 2000 [26]. Cube casts were used to make specimens measured 100 mm × 100 mm x 100 mm conforming to European codes, BS EN 12390–1:2012 [27]) and the specimens were cast conforming to BS EN 12390–2:2009 [28]. The target of the concrete mix was strength class C32/40 at approximate mix proportions of 1: 2: 3 (cement: sand: gravel).

The water content was substituted with digestate in the percentages of 0%, 25%, 50% and 100%. The 0% replacement was used as the control specimen and the mixes for the first stage of the study had a water-to-cement (hereafter referred to as 'w/c') ratio of 0.5. For the second stage, water was added at each substitution dosage of digestate until a consistent compactable mix was attained. The error was estimated by casting three cubes for each testing age and reported as an average. The cube casts were left within the moulds for 24 h before being stripped, marked, and submerged in a water tank at temperatures of 20 °C ± 2 until their testing age. As the digestate had a reducing effect on the workability of the cement the concrete slump height was controlled to a range of 10–20 mm by using an industrial standard synthetic carboxylated polymer modified superplasticiser, primarily for self-compacting concrete. The different modifiers were investigated across two different trials, and thus used different control specimens,

Table 1
Experimental set up of concrete mixes preparation.

Concrete Mix[a]	Cement (kg)	Fine Aggregate (kg)	Coarse Aggregate (kg)	PC Digestate (L)	Water (L)	With Plasticiser (ml)	With Additional Water (ml)
Control (0%)	2.5	5	7.5	0	1.25	0	0
25%	2.5	5	7.5	0.31	0.94	20	0
50%	2.5	5	7.5	0.63	0.63	20	300
100%	2.5	5	7.5	1.25	0	50	500

[a] Column indicates % of water replacement grade with PC digestate.

conducted approximately 4 weeks apart. A two-factor ANOVA in Design Expert (v.13) was conducted on the variable 'plasticiser' to investigate the impact of the presence or absence (additional water only) on compressive strengths at different water replacement grades with the variable '%Digestate', which also allowed to identify the interaction effects on performance (Fig. 1S–B in Supplementary material). The ANOVA ($\alpha = 0.01$) results are reported in Supplementary Tables 3S–5S, where factors levels were 2 (coded 'Yes' and 'No') and 4 (coded 'None', 'Low', 'Medium' and 'High') for the plasticiser and the %Digestate respectively, and the significance with a full fit summary (Table 6S) of the modified model for day 28 is also reported.

3. Results & discussion

3.1. Paper sludge composition

The PC was primarily composed of fibrous material with trace quantities of metal, plastic and glass impurities that accounted for less 0.20 wt% of the starting material (Supplementary Table 2S). The PC was characterized using several methods, as described by Table 2.

The fibrous material was primarily composed of cellulose and acid insoluble lignin, 20.3 wt% and 27.1 wt% respectively, that can be accounted for to the attributes of the individual paper recycling plant. Both cellulose and acid-insoluble lignin are mostly recalcitrant during AD and both have been previously utilized as concrete additives [29], though in far higher concentrations than those applied in this study.

Trace quantities of acid soluble lignin (0.6 wt%) were detected, which are indicative of Kraft process derived recycled paper pulp, however it should be noted that this value may be overestimated due to interference of inks and dyes with the acid soluble UV absorption spectra. The PC exhibited high ash content of 36.7 wt% which was mostly acid soluble, with the acid insoluble fraction being 4.9 wt%. The acid soluble mineral composition was estimated using nitric acid digestion showing a considerable calcium content in the PC followed by aluminium, iron and magnesium. Calcium carbonate is a common paper filler for improving paper brightness and durability, this is commonly filtered out during paper recycling due to sieve compatibility for small particle size.

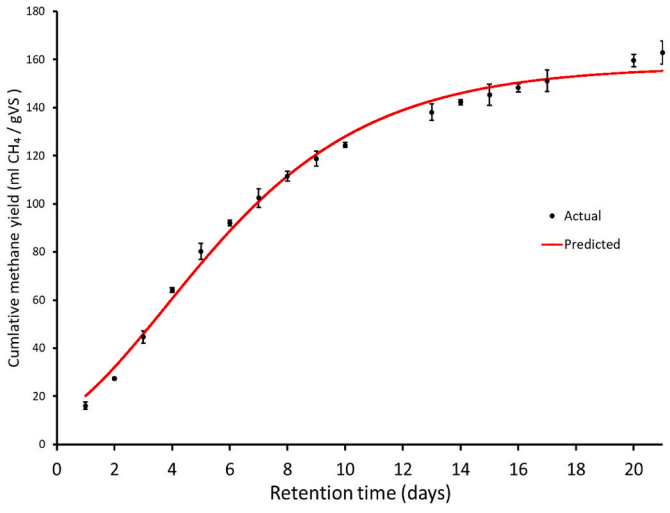

Fig. 1. Daily cumulative methane yield (ml CH_4/gVS) for PC during the 21-day digestion period.

Table 2
Compositional analysis of PC.

Parameter	%
Proximate Analysis	
Moisture (%wt.)	50.07
Total solids (TS) (%wt.)	49.93
Volatile Solids (VS) (%dwt.)	63.39%
Ash (%dwt.)	36.71
Contamination (%dwt.)	2.00
Elemental Analysis (%dwt.)	
C	34.20
H	4.44
N	1.05
O	23.60
C/N	32.6
Trace element Analysis (%dwt.)	
Na	0.13
Mg	0.36
Al	0.54
P	0.25
S	0.25
K	0.06
Ca	11.44
Cr	0.48
Mn	negligible
Fe	negligible
Co	0.01
Ni	0.03
Cu	0.02

The carbon nitrogen ratio of over 32 is outside the recommended ratio (15–25) for AD and the digestion will require a form of nitrogen supplementation (e.g. co-digestion) to improve and stabilize biogas (and methane) yields for commercial scale operations however, the presence of P, S and Fe would reduce the requirement for micronutrient supplementation. The PC would therefore require co-digestion at commercial scale for the nitrogen balancing. It should also be noted that the S, Al and Fe elements may be present in the form of common paper fillers gypsum and kaolinite.

3.2. Anaerobic digestion of PC

The 21-day BMP of PC under mesophilic conditions is shown in Fig. 1. The 21-day cumulative methane yield was 163 ml CH_4/gVS or 103 ml CH_4/gTS with an average biogas quality of 52.3 %CH_4. The results in this study are comparable to other works [30] that achieved yields in the range of 49–167 ml CH_4/gVS with similar feedstocks over 21 days. The biodegradability index was 47.7% compared with the theoretical maximum methane yield based on the elemental analysis, which suggests significant quantities of undigested material is remaining post digestion. The daily cumulative methane yields were successfully modelled with the Gompertz equation ($p = 0.990$) and shown in Fig. 1.

The Gompertz model parameters were derived, and the cumulative

methane yield estimated at 157.1 CH_4 mL/gVS with a maximum methane production R_{max} rate of 14.79 and a lag time λ of −0.09 days. Between the period of 15–21 days the daily production rate was ≈3 ml CH_4/gVS, which indicates the slow degradation of complex molecules was occurring. The contrasts between the high initial activity and minimal lag time strongly suggests that PC includes separate fractions that degrade over significantly different timeframes. The PC's extractable fraction was not analysed, though it could possibly include short water-soluble paper fibers that cause the high initial methane production rate; perhaps multiple methods should be undertaken to identify a more articulate and revealing kinetics model as it has been done for bioplastics degradation [31,32]. The overall result shows that the AD of PC is technically feasible with significant potential, but further work is required to optimise the AD conditions.

3.3. Digestate composition

The digestate was analysed for several compositional properties as shown in Table 3, which revealed the digestate has advantages for use in concrete such as neutral pH, low TS and VS content and extremely low concentrations of water-soluble ions (Cl^- and SO_4^{2-}) known to act as concrete's durability deteriorators.

Another advantage for use of digestate as water substitute for cement hydration is that pH was found at 7.65, in line with the normal AD operating parameters. Total solid percentage in digestate was 5.7 %wt., of which the majority were volatile solids (at 4.1 %wt.). The solid fraction contained significant quantities of nitrogen and sulphur, 5.1% and 0.6% respectively. This is due to the brewery inoculum source that would be significantly lower during the AD of pure PC. The analysis of soluble ions indicated significant quantities of sodium, potassium, and chloride with near negligible levels of sulphate and calcium ions and would be considered moderately saline. The chloride content in the sludge will corrode reinforced concrete and this, together with sulphur content, represents a general disadvantage for using wastewaters in concrete, however the low absolute content of 0.03 wt% of the digestate would be negligible as it is within standard ranges for ground water [33], which also indicates PC digestate would be suitable for concrete applications.

Table 3
Compositional analysis of the digestate from the 21-day AD of PC.

Digestate Properties	
Proximate Analysis	
pH	7.65
Total Solids (%wt.)	5.7
Volatile Solids (%wt.)	4.1
Ash (%wt.)	1.6
Elemental Analysis (%dwt.)	
C	38.7%
H	5.6%
N	5.1%
S	0.6%
O	21.9%
Ash	28.1%
Major Water-Soluble Ions (g/L)	
Na^+	1.279
Mg^{2+}	0.005
K^+	1.137
Ca^{2+}	0.053
PO_4^{2-}	0.035
Cl^-	0.325
SO_4^{2-}	0.085

3.4. Concrete analysis

3.4.1. Concrete with plasticiser

In the first part of the study the samples were prepared whereby the water content was substituted with PC digestate in percentages of 0%, 25%, 50%, and 100%. The 0% replacement also referred to as the 'control specimen' was used as the reference to which the performance of all replacements was measured. A constant water cement ratio (w/c) of 0.5 was used for all mixes, for comparability of results. Appropriate amounts of plasticiser were added (as reported in Table 1) to attain a consistent compactable mix. The results of compressive strength testing show a decrease in workability with increasing water substitution grades with PC digestate. Given the solid content of the digestate (approximately 6 wt%), the higher replacement grade would increase the solid content in the concrete mixture, hence would cause a greater requirement for plasticiser addition, as also evidenced by Fig. 1S (in Supplementary material). The anionic lignosulfonate plasticiser under normal circumstances helps disperse the flocculated cement particles, to form a colloid, through a mechanism of electrostatic repulsion. A key finding of this study for potential future use of PC digestate in concrete is that even at 100% water replacement, it is still possible to achieve a coherent and workable mix.

Fig. 2 reports the results of the characteristic compressive strengths at 7 and 28 days of hardened concrete with 0%, 25%, 50%, and 100% water replacement with PC digestate.

The compressive strength of any material is defined as the resistance to failure under the action of compressive forces. Especially for concrete, compressive strength is a key parameter to determine the performance of the material during service conditions. The characteristic strength is defined as the strength of the concrete below which not more than 5% of the test results are expected to fall as specified in the relevant British and European standards. The ANOVA test results (Tables 3S–5S) show overall across the curing period only the percentage of water substitution with digestate (%Digestate) and its interaction with plasticiser have a significant ($p < 0.01$) effect on performance. The results show that both the 25 and 50% samples are exhibiting a marginally higher strength than the control mix, this performed much higher that other digestates in the range of 17.1–20.3 MPa that used cow, cattle and poultry substrates [17] around ~0.5 w/c ratio. It is also worth noting both mixes exhibited excellent early age strengths of over 35 MPa within 7 days – both satisfying the C32/40 classification. C32/40 grade concrete is the standard used globally for many structures and civil engineering applications, such as house foundations, paving of outdoor terraces or garage floors. Although the mix at 100% replacement grade had a lower strength, however, it still exceeded 30 MPa after 90 days which is still suitable for numerous structural applications. The reduced

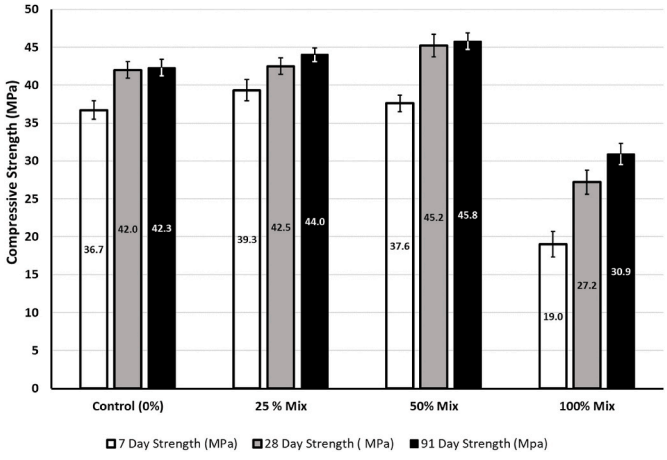

Fig. 2. Characteristic compressive strength of concrete with digestate replacement of water with plasticiser.

rate of strength gain over time with added digestate content suggests that the digestate acts as retardant, which has previously been reported for wastewaters [34]. In contrast with wastewaters however, PC digestated appears to perform better for the 25%–50% grades of substitution, as replacement with secondary and tertiary treated wastewater has been found not to reach 30 MPa of strength at day 28 [35]. Typically, this could be associated with the chemical effects of chloride and sulphite on compressive strength [36].).

3.4.2. Plasticiser free concrete

Similarly to the mixes described section 3.4.1 and as reported in the far-right column of Table 1, the water content was substituted with PC digestate in percentages of 0%, 25%, 50%, and 100%. However, in this case a tap water solution was incrementally added until a consistent workable and compactable mix was developed without any admixture (plasticiser) content. Thus, in this case the main objective was to ascertain the minimum hydration level achievable without an admixture – this is a useful indicator for design mix consideration. The minimum w/c ratios for each mix were 0.50 for both 0% and 25% degree of water substitution with digestate, 0.60 for 75% degree of water substitution with digestate and 0.65 for 100% degree of water substitution with digestate. As expected, the minimum achievable w/c ratio increases with water replacement grade. It is interesting to note at 25% digestate replacement has negligible effect on the workability. However, the ANOVA Tables 3S–5S in Supplementary show that above 25% degree of water substitution with digestate, performance results in reduced compressive strength ($p < 0.01$) that can be attributed to an increase in hydration demand. Similarly to previous findings that included use of plasticiser, we identify the cause in the high solids content in the concrete mix, which increases with digestate replacement grade.

The compressive strengths of the respective mixes at different grades of water substitution with PC digestate are reported in Fig. 3.

Any comparison of strength values must take into account the differing rate of hydration for the mixes; however, the findings are as expected and follow the trend as per rate of hydration of the cementitious compounds [18]. In fact, the best performing mixes exhibit up to ~7% extra strength compared with the respective control and are comparable to the compressive performance achieved when using wastewaters in concrete [37]. There is minimal difference between 28- and 91-day strengths, thus the 28-day strengths are indicative of the maximum achievable strengths at varying digestate replacement grades without any admixture addition. An interesting finding for the 100% digestate mix had a slightly higher strength at a higher w/c ratio without plasticiser than a lower w/c ratio with a plasticiser. This could be due to the higher digestate replacement grade, hydration is hindered and can partially be alleviated by an increase in w/c ratio. The results are quite promising with the 25% digestate mix satisfying the C32/40 classification; the higher grade of water substitution achieved compressive strength in excess of 30 MPa, thus all digestate mixes have potential for structural and several civil engineering applications as specified by the relevant British and European standards [18].

4. Conclusions

This study investigated the valorisation of recycled PC by sequential valorisation using AD to produce biogas and subsequent utilization of the digestate for concrete production. The mesophilic digestion of the PC produced 163 ml CH_4/gVS over 21 days and showed high initial activity that reduced significantly over time, due to the high cellulose and lignin contents. Coherent and workable mixes were achieved with all levels of sludge (digestate) content. The compressive strengths showed good repeatability, with strengths capable of structural applications being observed at 28 days, with all mixes with admixtures exhibiting impressive early age strengths at 7 days of over 30 MPa. These results show that paper digestate can be used as a potential water replacement, thereby enhancing the sustainability of concrete.

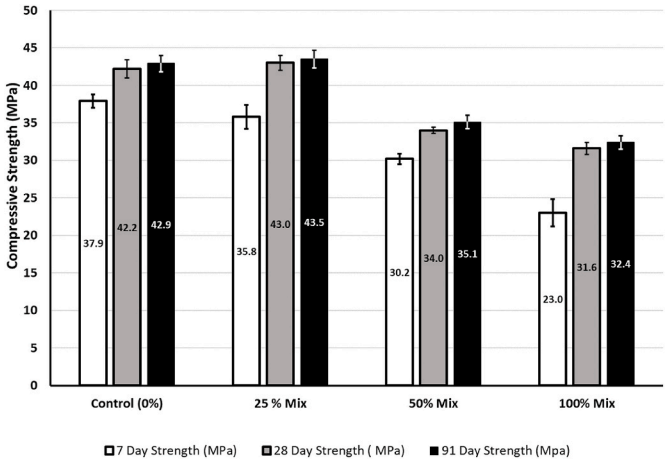

Fig. 3. Characteristic compressive strength of concrete with digestate replacement without plasticiser for minimum w/c ratio achievable without admixtures.

The findings suggest that the even dispersion of the digestate material through the concrete colloid has a critical effect on the concrete's compressive strength and should be investigated further. The overall results of this study show that the sequential valorisation of the paper waste was successful and that the use of digestate as a water replacement in concrete could lead to the valorisation of marginal organic wastes via AD, if the digestate is used in this way. Screening the compositional constituents for chlorine and sulphur contents is always recommended when assessing waste effluents and biomass aggregates as they are known to reduce concrete's durability. The compositional results suggest that paper crumb digestate is suitable to replace water for cement hydration, however this should be investigated further via durability tests that will ascertain the longevity of the concrete obtained from paper digestates.

CRediT authorship contribution statement

George Hurst: Formal analysis, Writing – original draft, Writing – review & editing. **Ash Ahmed:** Conceptualization, Formal analysis, Writing – original draft, Investigation, Methodology, Supervision, Writing – review & editing. **Steven Taylor:** Formal analysis, Investigation, Writing – review & editing. **Silvia Tedesco:** Conceptualization, Methodology, Data curation, Formal analysis, Funding acquisition, Investigation, Project administration, Writing – review & editing.

Declaration of competing interest

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: Silvia Tedesco reports financial support was provided by Engineering and Physical Sciences Research Council.

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Appendix A. Supplementary data

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