

## DEVELOPMENT OF A NOVEL GLYCEROL BOUND MASONRY UNIT COMPOSED ENTIRELY FROM ENVIRONMENTALLY FRIENDLY MATERIALS

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### ABSTRACT

This paper presents an investigation into the development of a novel glycerol bound masonry unit. The research is motivated by the desire to enhance the sustainability, both nationally and globally, of masonry units. The production of the units from entirely waste materials is possible because a blend of waste glycerol and waste cooking oil is used as the binder; the aggregates include incinerator bottom ash (IBA) and pulverised fuel ash (PFA). The compressive strength and water absorption properties of the units were measured to assess their performance in relation to masonry products found in the UK. These properties are good indicators of overall performance. Results from the study demonstrate that the units can be produced with properties that are at least equivalent to those of currently used masonry units.

**KEYWORDS:** masonry, glycerol, cooking oil, oxidation, hardening, strength

### INTRODUCTION

Although steps have been taken to improve the sustainability of traditional masonry binders, i.e., cement and clay, these binders can still, on the whole, be classed as unsustainable. Clay is a finite resource and the manufacture of cement is still responsible for 6,000,000 to 7,000,000 tonnes of CO<sub>2</sub> produced each year in the UK [1]. More sustainable binders are therefore required. Previously, it was shown that bitumen [2,3,4,5,6] and more recently waste cooking oil (WCO) could be used as a binder for masonry units [6,7,8]. In the latter work, 5% WCO was added to a mix of limestone aggregates and after the mix was moulded, compacted at 4MPa and cured for 12 to 48h at 160°C, compressive strengths between 14.2MPa to 30.7MPa were obtained. The limestone aggregates used in that investigation were traditional aggregates, excavated from a quarry and typical of those aggregates used in the manufacture of concrete. However, when waste aggregates were substituted for the traditional aggregates, the binder content increased to 12% [8]. The use of glycerol as a binder / transfer mechanism for the oil is seen as a way of reducing the required oil content.

This research attempts to combine a blend of waste glycerol and waste cooking oil binder with IBA and a waste filler material to produce a masonry product with qualities that are at least equal to current clay bricks or concrete blocks. Therefore, a novel, more sustainable masonry product will be developed, i.e., it will contain 100% waste materials and possess a smaller embodied energy. The major aim of this work is to develop the use of a combination of waste glycerol and waste cooking oil as binders for construction products. The objectives are:

- + To find a significant use for various solid waste types or by-products

- + To allow the more sustainable use / disposal of waste cooking oils
- + To allow the use / disposal of waste glycerol

The research looks at the performance of the new units in terms of compressive strength and water absorption properties as previously [6, 7, 8] it is found that these properties are good indicators of overall performance (i.e. long-term behaviour, durability etc).

## **MATERIALS, SAMPLE PRODUCTION AND TESTING METHODS**

Natural sand (<5mm) and gravel (5-10mm) were used as aggregates to produce the Control samples. All the natural aggregates were supplied by Tarmac Road stone, Wolverhampton, West Midlands, UK. The mixing ratio of natural sand to gravel was 70/30. This ratio was chosen as it was similar to the fine to coarse aggregate ratios used in the manufacture of the IBA samples (see below) with which the sand/gravel mix was compared.

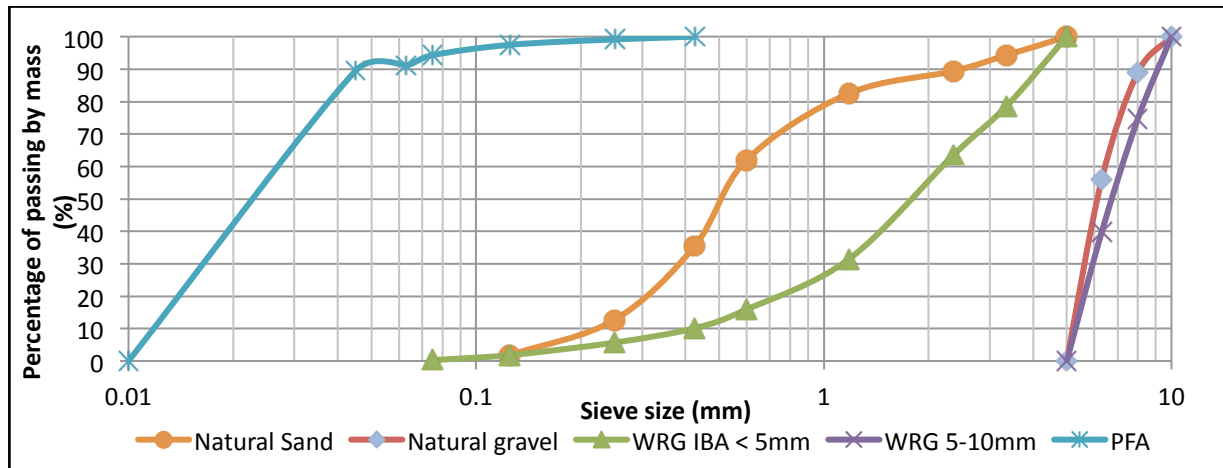
Incinerator Bottom Ash (IBA) used in this study was collected from Eastcroft Energy from Waste Facility - Waste Recycling Group (WRG) –Nottingham, UK. Two basic particle sizes of 5 – 10 mm and <5mm were chosen; 70% of the IBA aggregate was 5-10mm and 30% was <5mm. In order to maintain consistency, this mixing ratio was used throughout the whole research project. This ratio is also close to the ‘natural’ ratios found in the collected IBA material.

Pulverised fuel ash (PFA) was used as a filler to the mix containing either IBA or natural aggregates. It was collected from FerryBridge ‘C’ Power Station – West Yorkshire. The specification for PFA complies with the British Standard EN 450 Type S [9] with more than 80% of the particles less than 45microns. Typical properties of the aggregates used in this investigation can be found in

**Figure 1: Gradation of the aggregates used in the investigation**

**Table 1. Error! Reference source not found.** shows the gradation of the aggregates used in the investigation.

The binder used in this investigation was a blended binder composed of glycerol and cooking oil with the mixing ratio of 3:1 by weight [10]. Pure glycerol was ordered from ReAgent Company, Cheshire, UK. The clean and waste cooking oil was ordered or collected from Leeds University Catering Service and which originally came from the KTC Company (Wednesbury, West Midland, UK). Waste glycerol was collected from the Greenegy Biodiesel production plant in Immingham, South Humberside, UK.



*Mix 3 - Waste binder samples - with waste aggregate (100% waste materials)*

The aggregate and binder composition was the same as with the pure binder units (Mix 2) except that the binder was a mix of waste cooking oil and waste glycerol.

When mixing completed, the material was weighed and then put into the mould and manually compacted using a trowel to ensure all the sample's corners were filled properly. The material was divided into 2 or 3 portions and placed into the mould in two or three stages. The sample cross-section was 100 x 100mm, with a total amount of material of 1.0 – 1.3kg used depending on its required thickness/depth which was between 50 to 70mm.

After placing the material in the mould, the sample was compacted using a 500kN capacity press machine. Compaction levels of 1, 2, 4, 8, 16, 20, 24, and 32MPa were trialled. It was found that with a compaction pressure of 1MPa, the samples needed to be handled carefully, so it was recommended that the chosen compaction level should not be under 1MPa. The most effective compaction level was determined at 8MPa.

Curing temperatures of 140, 160, 180 and 200°C were chosen and applied using a convection oven. Curing was conducted to allow the binders to harden by oxidation and to allow any chemical reactions between the binders, and between the binders and the aggregates to occur. Different curing durations were selected namely 24, 48, 72, 96 and 120h in order to evaluate the effect of curing time on the strength development of the samples.

Samples were laid vertically on the steel grid shelf of the oven with reasonable spacing to allow the hot air to flow freely around the samples. This ensured that the surface of the samples were sufficiently open to the oxygen in the air in the oven; it is believed that the oxygen plays an important role in binder hardening (oxidation) which helps create the strength of the samples [8]. Curing temperatures were controlled to  $\pm 2^\circ\text{C}$

After curing, the samples were taken out and left at room temperature for at least 6 hours to cool down before being ready for testing. The samples were weighed at the time of taking out of the oven and before crushing. Thickness measurements were taken using digital callipers accurate to 0.01mm.

To confirm, the latter three types of samples (Mix 1, 2 and 3 mentioned above) were compacted at 8MPa compaction pressure. Curing temperature was selected as 160°C and the curing duration was 96hours. These parameters were chosen as they produced samples with maximum strength. However, the lower strengths for shorter curing durations and lower temperatures also produced units with acceptable properties (see below). One of the benefits of this manufacturing process is that units can be produced with properties which are more in-line with the required in-use property, i.e. currently traditional clay units can be produced to achieve certain levels of frost resistance but in doing so possess strengths far in excess of what would be required in practice. Figure 2 shows a brick and a block sample after manufacturing.



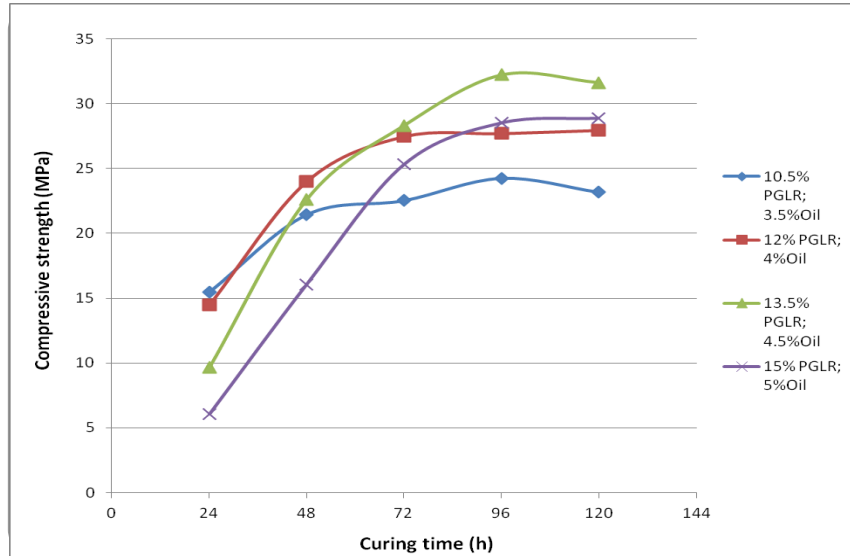
**Figure 2 Brick and block samples**

## TEST RESULTS

**Surface of the samples:** Although uncured (green) samples had low strength and required careful handling, after being cured, they possessed higher strengths and could thus be more easily handled. The final products also had smooth and flat surfaces with sharp arises.

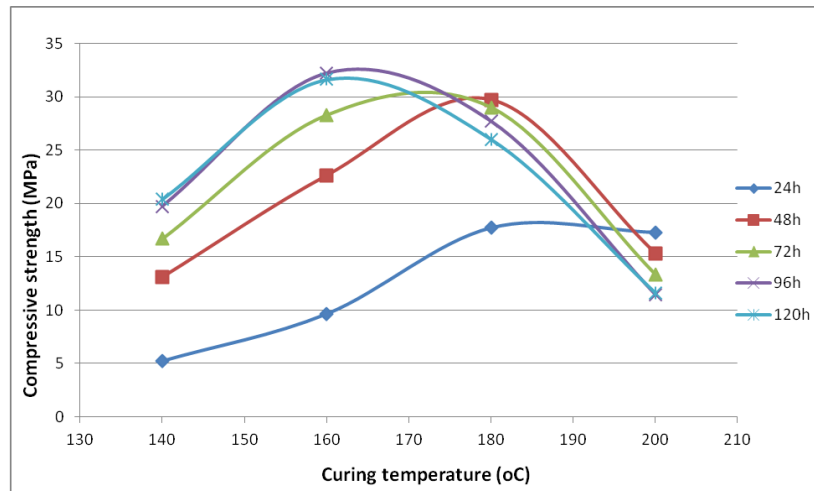
**Bulk density:** Bulk density was measured by water replacement and was found as expected to be influenced by the compaction pressure. The bulk densities obtained for the cured waste and pure binder samples ranged from 1.64 to 1.83 g/cm<sup>3</sup>. For control sample (Mix 1), the density was 1.91g/cm<sup>3</sup> which were greater than the density of Mix 2 and Mix 3 samples. This is thought to be due to the lower porosity of IBA aggregate which produces the lower density and therefore better manual handling properties.

**Compressive strength:** Compressive strength tests were carried out using a 3000kN compaction capacity testing machine. The testing procedure complied with the British Standard document BS EN 12390-3: 2002 [11]. Samples were produced with dimensions of 100x100x55mm. The loading rate was selected from between 0.2 to 0.6MPa/s depending on the roughly predicted strength of the samples. Figure 3 shows that, at 160°C curing temperature, the samples' compressive strength increased gradually between 24 to 96 hours of curing and levelled off at about 24 to 32MPa depending on the binder content. For the total blended binder content of 14 and 16%, the compressive strength levelled off at around 72hours, with strength values of about 24 and 27.5MPa, respectively. The highest compressive strength obtained was 32.22MPa with a curing time of 96 hours and 18% total binder content, this was also the highest value for the whole testing programme. The lowest compressive strength obtained was 6.05MPa with a curing time of 24 hours and 20% total binder content.



**Figure 3: Compressive strength (MPa) vs. Curing time (h) at 160°C curing temperature**

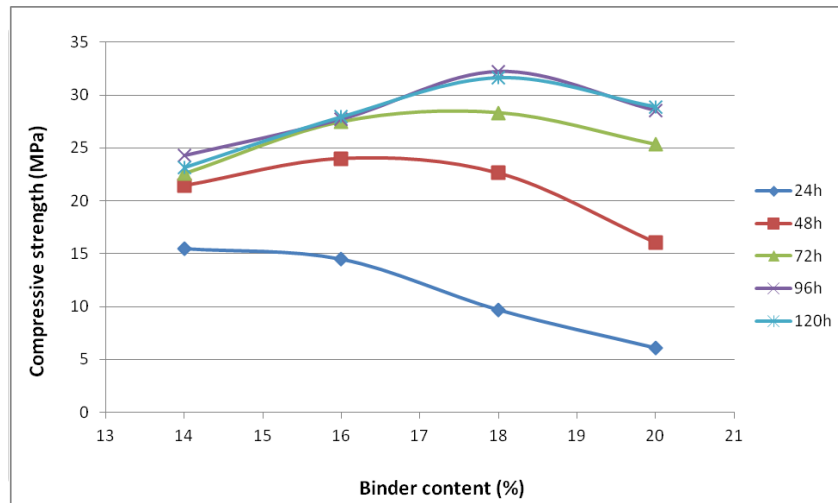
Curing temperature is one of the major factors (see Figure 4) which affects the strength development of the samples. In most cases, for binder contents and different curing durations, the compressive strength of the samples increased when the curing temperature increased from 140 to 160 or 180°C. The strength then decreased when the curing temperature increased to 200°C. This is clearly shown in Figure 3. It can be seen that the position of the highest compressive strength value for all binder contents is located in the middle of the surveyed boundaries. Therefore, it can be concluded that the optimal curing temperature is in the range of 160 and 180°C.



**Figure 4: Compressive strength (MPa) vs. Curing temperature (°C) for samples with 18% total binder content**

Figure 5 shows that, for most cases, the compressive strength of the samples increased when the total binder content increased from 14 to 16 or 18%. Beyond 18%, the strength decreased. There were some exceptions to this, i.e. where there are low curing temperatures and short curing time

durations. In these exceptional cases, the effect of the heat was insufficient to fully cure the samples.



**Figure 5: Compressive strength (MPa) vs. Binder content (%) at 160°C curing temperature**  
 After the optimisation study, the highest compressive strength samples were made using 160°C curing temperature, 96h curing time and 18% total binder content. These values were used to produce samples with waste binders (waste cooking oil and waste glycerol) which were then compared with control samples made from natural aggregates and pure binders.

From Table 2, it can be seen that the compressive strength of the pure binder samples with waste aggregate (Mix 2) was 87.5% of the compressive strength of the control samples made with natural aggregate (Mix 1). It is thought that this reduction in strength may be caused by the strength and structure of the IBA particles as they contain a larger amount of voids and IBA was not as strong as the natural gravel. The high porosity of the IBA also lead to a higher amount of binder being required, i.e. 18% total binder content for pure binder samples with IBA and 13.3% for the control samples with natural aggregate.

**Table 2: Compressive strength of control sample, pure binder and waste binder samples**

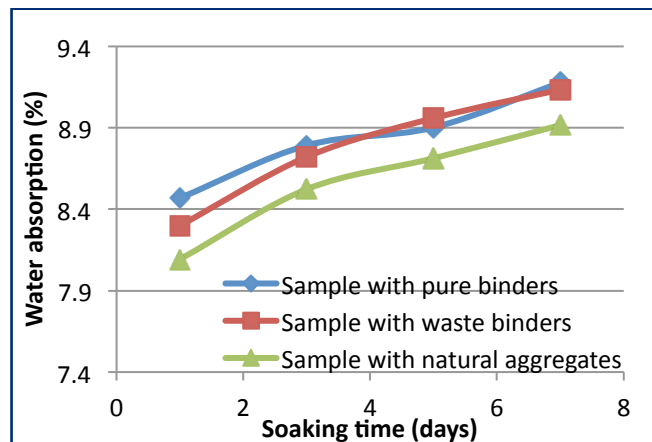
Properties	Natural aggregates (Mix 1)	Pure binders (Mix 2)	Waste binders (Mix 3)	Recommended value
Compressive strength (MPa)	38.4	33.6	31.3	$\geq 5$ for Damp proof course 1,2 [13]
Water absorption (%)	7.26	8.03	8.15	No limits for other types of bricks [13]
Initial rate of water absorption (kg/m <sup>2</sup> .min)	0.067	0.117	0.083	$\leq 1.5$ [14]

Table 2 also shows that the compressive strength of the waste binder samples (Mix 3) was about 93.1% and 81.5%, respectively, of the pure binder samples (Mix 2) and the control sample with

natural aggregates (Mix 1). However, the compressive strength of all these samples was relatively high and much greater than the required compressive strength of current commercial concrete blocks used in the UK [12]. Mix 3 and Mix 2 contained the same amount of aggregates and binder. However, the binder used for Mix 3 was the mix of waste glycerol (which contained up to 92% glycerol [10]) and waste cooking oil. The reduction in compressive strength of Mix 3 in comparison with Mix 2 is thought to be due to the presence of impurities that reduced the percentage of effective glycerol.

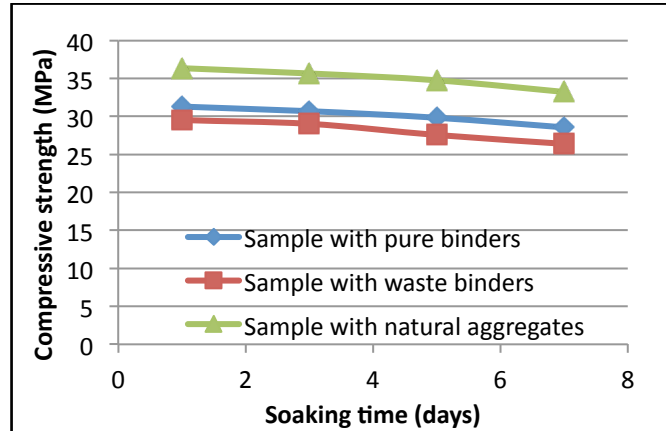
**Water absorption and water stability:** The water absorption test for all the samples in this investigation was in accordance with British Standard document BS EN 771 - 1: 2003 – Annex C [15]. The method used was the 24h cold water immersion test. In addition to the 24hour method, the samples were also soaked for a longer period of time, namely 3, 5 and 7 days, to evaluate the water stability/reactivity of the binder.

Figure 6 and Figure 7 show the changes in water absorption and compressive strength of the different samples with the different soaking times, respectively. The water absorption of the samples increased when the soaking time increased. The water absorption of the samples containing natural aggregates was slightly lower than that of the pure binder (Mix 2) and the waste binder (Mix 3). In general, the water absorption of all samples was between 8.1 to 9.2%. Figure 6 also shows that samples are still absorbing water after 7 days. This is thought to be due to the fact that glycerol was being used as part of the blended binder. The blended binder formed a thin coating layer on the outside the aggregate and filler particles. This thin layer of binder will initially prevent the water from being absorbed by the aggregates because 1) it provides a physical barrier and 2) glycerol is hydrophilic and will therefore ‘attract’ the water itself. This is thought to explain why it takes longer for the sample to become fully saturated. An observation of the failed samples, after being crushed showed that after one to three days of immersion, a ‘dry core’ was still present in these samples confirming that the water had not been fully absorbed into the samples.



**Figure 6: Water absorption (%) vs. Soaking time (days) for different samples**





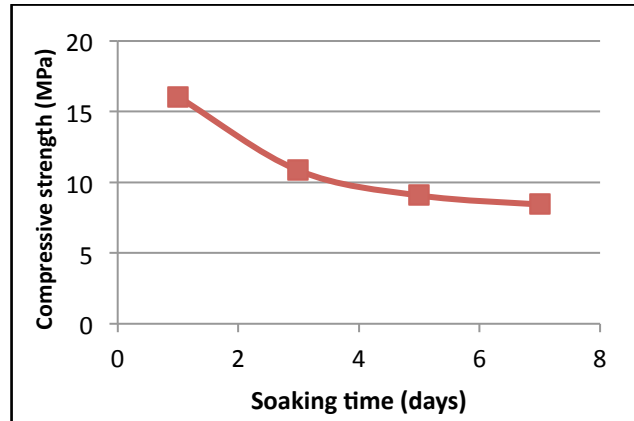
**Figure 7: Compressive strength (MPa) vs. Soaking time (days) for different samples**

From Figure 7, it can be seen that the compressive strength of the Mix 2 samples (pure binder) and Mix 3 (waste binder), after 1 and 7 days of soaking and then drying for 48 hours at 105°C, was 29.5 and 26.4 MPa, respectively. These strengths after soaking were 5.75 and 15.65% less than their original compressive strength. Similarly, the compressive strength of the pure binder samples (Mix 2) was from 28.57 to 31.27 MPa and that of samples with natural aggregate (Mix 1) was from 33.23 to 36.33 MPa. These values were still greater than 85% of the original compressive strength of each of the sample types. The decrease in the samples' compressive strength was not as high as expected. The small reduction is thought to be due to the fact that the samples were dried prior to strength testing. Even drying at 105 degrees C appeared to have restored some of the bonding within the matrix (i.e. the water is believed to have diluted the bonding effect of the glycerol, however, when it is removed by drying the glycerol will bind the aggregate once more).

To investigate this further, additional samples were immersed in water and strength tested without drying. This time they exhibited much lower compressive strengths after 1 to 7 days of soaking (see Figure 8). It is believed that the penetration of water into the samples neutralises the polarised bonding of the cured glycerol with the aggregates by adding the  $H^+$  and  $OH^-$  from the water to the polarised glycerol molecule [10]. This dissolution of hydrophilic glycerol into the soaking water is thought to contribute to the decrease in compressive strength of sample. The consequential leaching with time of some of the glycerol from inside the unit may also explain the slow and prolonged increase in water absorption mentioned above, as the aggregates become exposed and subsequently saturated during the soaking process.

Figure 8 shows the change in saturated compressive strength of waste binder samples after soaking from 1 to 7 days. The saturated compressive strength decreased from 16 MPa to 8.1 MPa when the samples were soaked for 1 to 7 days. These strengths were equivalent to about 25 to 50% of the original sample's compressive strength. The decrease in saturated compressive strength was expected. Traditional clay and concrete blocks exhibit lower strengths when saturated with water due to the modulus of the water in the pores and the internal negative pressure systems this creates within the units (when unsaturated, the pores are filled with air of effectively zero modulus). However, the loss in strength is also thought to be due to the neutralising effect of the water and the possible loss of the hydrophilic glycerol binder which

was leached out by the soaking water. After 7 days soaking, the saturated compressive strength of the waste binder samples was still greater than the compressive strength of currently used concrete blocks in the UK [12]. Overall, these tests indicated that these samples were sufficiently water stable.



**Figure 8: Saturated compressive strength of waste binder sample (MPa) with different soaking time (days)**

**Initial rate of water absorption:** The initial rate of suction (IRWA) was determined in accordance with British Standard document BS EN 772-11:2000 [16]. Error! Reference source not found. shows that the average IRWA values of samples were from 0.067 to 0.117 kg/m<sup>2</sup>.min. It can also be seen that the IRWA of the control samples (Mix 1) was smaller than those of the pure binder and waste binder samples. This could be explained by the higher porosity of the IBA (or PFA) aggregate enhancing the samples' water absorption. However, these values were well within the maximum limit of 1.50 kg/m<sup>2</sup>.min recommended in BS5628 Part 3 [14].

Results from an investigation carried out by Dong [17] showed that the initial rate of water absorption of samples produced using a bitumen binder with different types of aggregates and filler was less than 0.05 kg/m<sup>2</sup>.min. Also, the water absorption of these samples was less than 6%. These values were smaller than those from this investigation in which hydrophilic glycerol was used as one of the binder constituents. Therefore, it is thought that the dehydrated glycerol [10] was the cause for the higher IRWA and water absorption in these samples.

## CONCLUSION

Samples bound with a blend of glycerol and oil can be produced with a level of performance that is at least equivalent to those of current traditional UK masonry units. The product appears to have a relatively high compressive strength (more than 30MPa) and even the saturated compressive strength is 8.1MPa which is greater than the compressive strength of normal concrete blocks. It is suggested that, even in the worst case of being submerged, the strength of these units is still favourable. Leaching of glycerol may be an issue and needs further investigation.

The units developed in this investigation are, in terms of sustainability, quite promising as the units will allow the utilisation of a large amount of waste materials such as incinerator bottom

ash, pulverised fuel ash, waste glycerol and waste cooking oil. Moreover, the curing temperature is low at 160°C, which helps to reduce the energy required for manufacturing (although shorter curing durations should be used).

Samples produced from waste binder possessed a slightly lower compressive strength (7%) than those produced using pure binder although the glycerol content found in the waste binder was only actually 92%. A comparison of the other properties of the two sample types (waste and pure binder) indicated that they are quite similar in performance. It therefore appears that the use of waste binder was highly effective and that the impurities in waste cooking oil and waste glycerol are insignificant with respect to the sample performance.

## ACKNOWLEDGEMENTS

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