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Quality inspection on recycled coarse aggregates using laser-induced breakdown spectroscopy

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Abstract Single-shot LIBS was applied for inline quality inspection of RCA (4–12 mm) transported on a conveyor belt at 0.5 m/s and 30 tonnes/hour under industrial operational conditions. Major granular contaminants, e.g. brick, gypsum, rebar, plastic and wood were classified of which classification errors were within the regulated contamination limits (e.g. 0.2–2 cm^3/kg for float and 1–5 *wt*% for sink contaminants). To this end, we first designed a LIBS prototype (without auto-focusing) with a large depth field (± 4 mm) complying with the stream surface height fluctuations to increase the single-shot sampling rate up to 95 %. Second, PLS-DA classification model was validated using manually prepared material batches. The quality (cm^3/kg or *wt*%) of new concrete cast using these material batches, e.g. mechanical strengths were correlated with the LIBS data (number of classified LIBS spectra). It is shown that the LIBS technology is potentially capable of providing efficient, automated real-time quality inspection on RCA in-situ.

Keywords: LIBS, single-shot, inline, classification, recycling, aggregate.

1 Introduction

Construction and demolition waste (C&DW) is characterized by high volumes (> 300 Mt/year in EU27 in 2012 [1]) at low material value per

[1] Eurostat

unit mass. In general, the profit margins in industrial C&DW recycling and the markets for the recyclable materials are under pressure [2]. This may largely be attributed to the fact that the quality of the recovered materials by the traditional industrial recycling processes is uncertain at best. This applies especially to RCA that can potentially be reused in the manufacturing of new concrete, which secondary materials are the main target in this work. The way forward to a successful concrete recycling operation is to maintain high throughputs in processing while assuring a consistent RCA product quality that can be proven to comply with accepted standards. This strategy will lead to higher volumes of RCA's to become available as cost effective, high-quality secondary building materials for new concrete production.

To realise the strategy, reliable and efficient quality control technology is required in-situ, i.e. in conjunction with the primary process. The control technology must automatically and continuously check the product stream of concrete aggregates for levels of cross-contamination that are left from the building demolition process and are not properly mitigated by the specialised aggregate cleaning technologies. However, to date, the lack of efficient inline quality inspection capability has been a major obstacle.

In this study, a sensor platform based on LIBS was developed and demonstrated for inline quality inspection on RCA at industrial scale. Material batches with known contamination levels were manually prepared to test if the partial least squares discriminant analysis (PLS-DA) model can achieve classification errors of major contaminants within their contamination limits according to the standard. Moreover, the relation between the LIBS and the quality data of the new concrete cast using these RCA batches was investigated.

2 Inline LIBS prototype-integration into the closed-loop recycling of RCA

Similar to the LIBS setup in [1], Fig. 6.1 shows the schematic of LIBS sensor platform designed for inline inspection of the 4-12 mm RCA.

[2] In the Netherlands, recycled concrete aggregates are sold for 12 euro /tonne on average to replace primary river gravel in new concrete. The processing costs are about 8-10 euro /ton and material transport adds 0.10-0.15 euro /ton/km.

The surface roughness of RCA transported on the conveyor is determined by a laser triangulation sensor as $\pm$ 4 mm. An optimised mismatch between the focuses of the laser and the parabolic mirror enables enough depth of view to prevent the need for an auto-focusing lens system. The Nd:YAG 1064 nm laser fires at the maximum repetition rate of 100 Hz at a fixed point perpendicular to the belt to ablate a small amount of material from the aggregate that passes the focus of the laser lens. This produces a tiny cloud of plasma ($\sim$ 1mm diameter) just above the aggregate surface. This very short-lived plasma (microseconds) cools down and transmits light with a spectrum that is characteristic for the material. After implementing a supervised classification algorithm using a reference LIBS spectral database, the acquired LIBS spectrum uniquely identifies the material at hand.

Still, several challenges had to be addressed to bridge the gap between laboratory research into LIBS that lead to an industrial adaptation towards a robust and compliant LIBS platform for inline inspection of RCA. The LIBS prototype was shielded and encased to protect it from weather influences and ADR dust as shown in the bottom-left inset in the inset of Fig. 6.1. The platform was set up in mechanical isolation from the ADR machine that produced quite heavy vibrations by being mounted on top of a stack of large concrete blocks resting directly on the ground. The incident laser beam and plasma light collection unit were guided by a sealed metal tube onto the RCA stream on the conveyor belt. This laser tube also provided a safe working environment for the personnel in accordance with class 4 laser safety regulation. A fast camera was integrated into the LIBS platform to be able to monitor the amount of material on the conveyor belt (belt speed $\pm$ 0.5 m/s) and to check the laser-optics settings. The inline LIBS prototype platform, consisting of hard- and software, has been demonstrated in Hoorn, the Netherlands on 10th June 2016 to collect and analyse LIBS data in a real-scale industrial concrete recycling plant, and was operated above the ADR coarse output (4-12 mm) conveyor belt that carried 30 tons of RCA per hour [3].

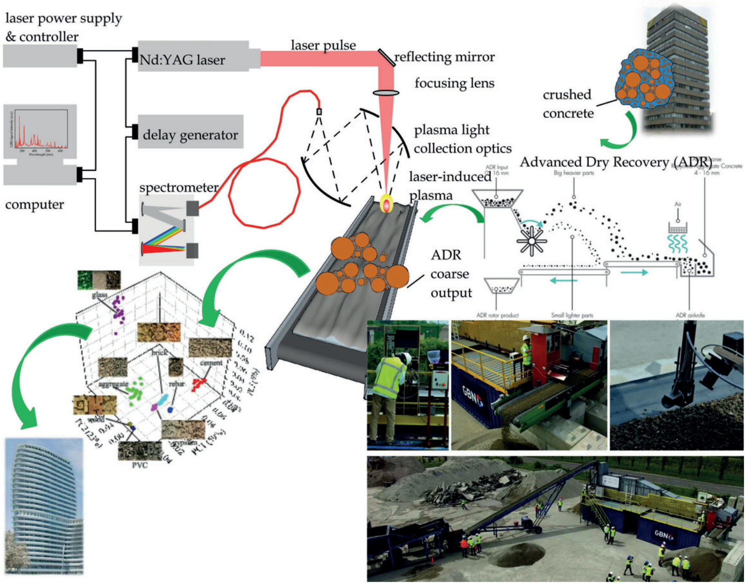

Figure 6.1: Principle and deployment of the inline LIBS platform into the closed-loop recycling of RCA. Inset photos: installation and implementation of inline LIBS sensor platform in Hoorn, the Netherlands on 10th June 2016.

3 Results and discussions

In parallel to the in-situ demonstrations, special batches of recycled aggregates (cf. Tab. 6.1 and Fig. 6.3a) were prepared and inspected using this LIBS platform. Several RCA batches were prepared as follows: 300 kg demolition concrete (0-44 mm) was sieved manually to 4-16 mm to obtain 160 kg materials. This was equal-partitioned into 4 fractions of 40 kg according to the European standard EN 932-2 [2]. Of each of these fractions, 10 kg was subjected to handpicking to determine the material composition according to the European EN standard 933-11 [3]. The major components in the sink fraction (0.63 ± 0.23 $wt\%$) were brick, glass, PVC, rebar and gypsum. The major components in the float fraction (1.29 ± 0.24 cm^3/kg) were wood and foam. The re-

[3] Video available at: https://www.youtube.com/watch?v=lHp3G-lOoOs

Table 6.1: Five sets of self-made RCA (4-16 mm, 30 kg) with varying contamination levels.

#Sample	Sink $[wt\%]$	Float $[cm^3 kg^{-1}]$	Comments
1 Reference	0.63 ± 0.23	1.29 ± 0.24	Recycled aggregate
2 Low sink, high float	< 1	2	Add extra 30 cm^3 float
3 High sink, high float	5	2	Add extra 30 cm^3 float and 1.5 kg sink
4 High sink, low float	5	< 0.2	Clean the float, add 1.5 kg extra sink
5 Low sink, low float	1	< 0.2	Clean the sink and float, add 0.3 kg gypsum

Table 6.2: PLS-DA classification errors using 15 components.

	PVC	brick	RCA	glass	gypsum	foam	wood	rebar
PVC	678	0	0	0	0	0	0	0
brick	0	676	0	0	0	0	0	2
RCA	0	0	678	0	0	0	0	0
glass	0	2	0	676	0	0	0	0
gypsum	0	0	1	0	677	0	0	0
foam	0	1	0	0	0	676	1	0
wood	1	0	0	0	0	3	674	0
rebar	0	0	0	0	0	2	0	676

maining 30 kg of each of the four fractions were modified manually as shown in Tab. 6.1 to improve the detectability of pollutant levels with LIBS and also to enhance the likely correlation with the quality of the produced new concrete samples.

3.1 PLS-DA classification model

We used a PLS-DA model [4][4] to determine the sensitive LIBS parameters by minimising the classification errors. To this end, we collected training datasets (i.e. LIBS data from reference aggregate batches and materials with well-known material composition) by selectively intro-

[4] using plsregress function, MathWorks® Matlab 2015b

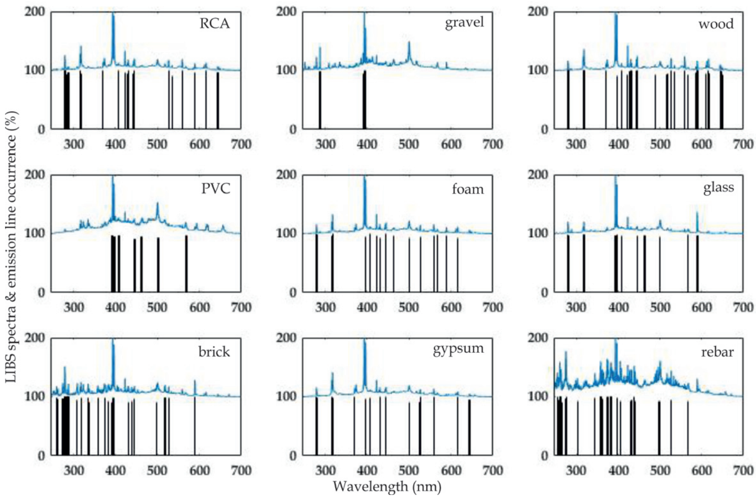

Figure 6.2: LIBS spectra (normalised to maximum as 100% and the occurrence of emission lines [%] observed for RCA, natural gravel (as a reference), and various pollutants handpicked from the concrete aggregates.

ducing more data variations, such as using larger optics-to-sample-surface distances or adding more material types. Each single-shot LIBS spectrum was normalised to its integral over the full wavelength range [5]. LIBS measurements were repeated three times per material batch, and each time 6000 single shot LIBS spectra were acquired at a rate of 100 per second while the laboratory conveyor belt shown was feeding at 30 kg/minute. Their raw LIBS spectra were shown in Fig. 6.2, in which emission lines with larger spectral occurrence $> 95\,\%$ (ratio of the number of spectra with identifiable emission line to the total spectra) were also shown underneath each LIBS spectrum. The emission line determination was described in [5]. It is noted that RCA is a new type of material of which the LIBS spectrum is more Ca-line-rich in comparison with that of gravel. During the collection of training data, we purposely introduced sample surface fluctuations larger than $\pm 20\ mm$ to emulate the fluctuations observed under real-scale operations. The multi-classification PLS-DA model was trained using 60 components which results in a mean-square error of 0.104 with 5-fold

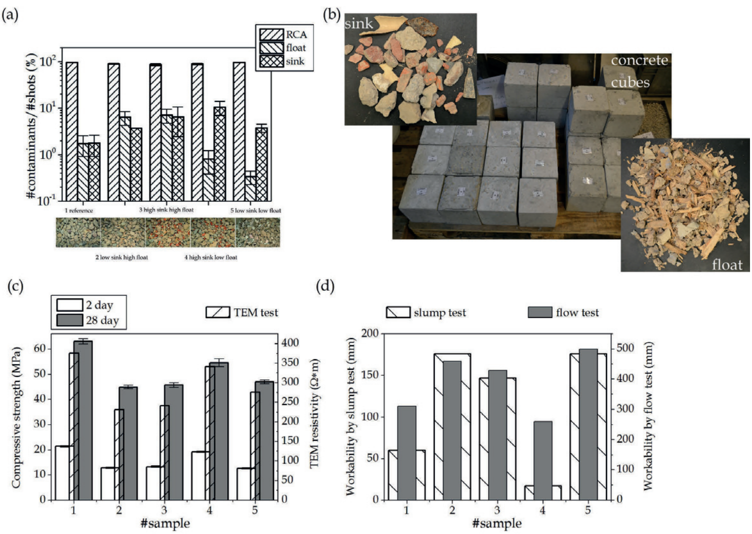

Figure 6.3: (a): Number of contaminants identified by LIBS, normalised to the total number of identified spectra. Results are for the five batches specified in Tab. 6.1, as shown in the photos underneath. The sink fraction contained PVC, brick, glass, gypsum, and rebar, while the floats contained wood and foam. The error bar represents 1STD, related to three duplicate measurements. (b): New concrete cubes cast using forenamed material batches and inherent sink and float contaminants. Quality tests on concrete cubes using five material batches (c): mechanical strength and (d): workability tests.

cross-validation. The classification errors are presented in Tab. 6.2. It is noted that the false-positive and false-negative errors for wood particles are 0.15 % (foam) and 0.59 %, respectively.

3.2 Relating LIBS parameters to the achieved quality of the new concrete

We first determine the contamination levels including composition and contents of the RCA's (4-16 mm) using LIBS. The results are shown in Fig. 6.3a as percentages of classified spectra, which agree rather well with the known material compositions. It is noted that the 1 % count ratio of floats corresponds to 2 cm^3/kg float contaminants and 1 % count ratio of sink materials corresponds to 1 wt% sink contaminants. The last two batches were cleaned using a sink-float separation before adding extra contaminants to reach the desired contaminant composition. Therefore, the float contents in those batches are lower than the float contents of the original coarse aggregates batch from which they were derived.

We then investigate the influence of contamination levels on the quality of the newly produced concrete cubes shown in Fig. 6.3b. The classified number of shots for each type of material proved proportional to the particle size, regardless of its specific shape. Furthermore, the number of particles can simply be transferred into contents (*wt*%) or volume percentages (cm^3/kg) since the average mass densities of all the materials are known.

To test the quality of the concrete cubes the following quality parameters were selected: 2 day and 28 day mechanical strengths were tested in the ENCI quality assessment lab [5], the electrical resistivity was measured using a two-electrode method (TEM), and the workability was determined using slump and flow tests. The recipe used to produce the C30/37 XC3 F4 class strength concrete was as follows: a mixture of cement using CEM I 52.5 N (80 kg/m^3) and CEM III/B 42.5 N LH HS/SR (240 kg/m^3); 5 batches of coarse aggregate (915 kg/m^3); river sand (805 kg/m^3); water-to-cement ratio of 0.54, and super-plasticizer (1.92 kg/m^3). Per batch 30 kg was used for 6 test cubes (150 mm) to determine the workability and mechanical strength (Fig. 6.3c). The error bar represents one standard deviation as obtained from three concrete cubes per type of test. The white bar indicates the 2-day compressive strength; the dark grey bar the 28-day compressive strength and the dashed bar the resistivity using TEM. In the workability tests (Fig. 6.3d), the dashed bar indicates the slump workability, and the light grey bar the flow workability. It is clearly shown that the trends of the 2- and 28-day mechanical strength and TEM values are similar. The resistivity may be assumed inverse proportional to chlorides ingress rate at room temperature [6]. The reference sample shows the highest strength and largest resistivity. The high-sink, low-float batch shows the second best performance, where the brick is the major sink contaminant. This in-

[5] Company webpage: `http://www.enci.nl/nl`

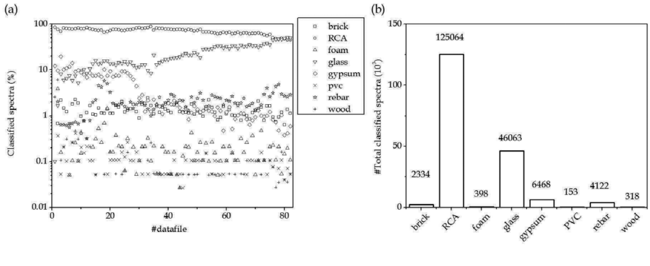

Figure 6.4: (a): Percentages of classified contaminants of each sampling burst in RCA over time. (b): Total number of classified contaminants in RCA.

dicates that brick degrades the strength and resistivity only to a small degree. The low-sink, low-float batch consisting of 1 wt% gypsum indicates that gypsum affects the concrete strength and resistivity more than brick, despite the fact that the gypsum contents were lower. Compared with brick and aggregate, gypsum is more brittle and its breaks down easily during the mixing procedure for making cement paste. According to its high workability, the gypsum batch may absorb less water. The second and third batch show similar strength and resistivity, indicating that the float contaminants are the major components that degrade concrete performance. They may also reduce water absorption when looking at the high workability.

3.3 Inline LIBS classification results on RCA

During the Hoorn demonstration, 60 tonnes of coarse aggregate products was processed by the ADR and inspected by the LIBS platform. Fig. 6.4a shows the classified numbers of LIBS spectra [%] in real-time over 82 sampling bursts. In total, 195235 LIBS single-shot spectra were collected of which 184920 shots could be properly classified, amounting to a 95 % success rate in LIBS sampling. As indicated in Fig. 6.4b), 125064 were classified as concrete aggregates, 2334 as brick, 6468 as gypsum, 4122 as steel rebar, 398 as foam, 318 as wood and 153 as PVC plastic. Using the previously determined correspondence between 1 % LIBS classification percentage and 1 cm^3/kg concentration of float contaminant, and taking wood as the most critical float contaminant in the

RCA market, the identified particles content accounted for 0.172 % of all particles which corresponds to a wood volume contamination level of < 0.2 cm^3/kg of aggregate product. This level complies with category $FL_{0.2}$ according to European-EN standards [3,7] for floating pollutants in concrete. It is remarked that this level complies accurately with the handpicking and float analysis results for a 10 kg sampler from the demonstration site.

4 Summary

In this present work, a LIBS prototype was set up for RCA recycling that achieved a sampling rate of 95 % by virtue of its long depth of field, corresponding to the surface roughness of RCA ($\pm$ 4 mm). Using normalised LIBS spectra as inputs, a PLS-DA multi-classification model was trained and validated on manually prepared RCA batches with different contamination levels (1-5 wt% for sink and 0.2-2 cm^3/kg for float contaminants). Same batches were then employed to cast into the new concrete, and their quality data were well-correlated with their LIBS classification results. Finally, this LIBS technology was employed for inline quality inspection of RCA (4–12 mm) at industrial scale (throughput of 30 tonnes per hour at transport speed of 0.5 m/s).

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References

1. H. Xia and M. Bakker, "Reliable classification of moving waste materials with *libs* in concrete recycling," *Talanta*, vol. 120, pp. 239–247, 2014. [Online]. Available: http://www.sciencedirect.com/science/article/pii/S0039914013009703
2. *European Committee for Standardization, EN 932-2: Tests for general properties of aggregates - Part 2: Methods for reducing laboratory samples*, Std., 1999.

3. *European Committee for Standardization, EN 933-11: Tests for Geometrical Properties of Aggregates - Part 11: Classification Test for the Constituents of Coarse Recycled Aggregate*, Std., 2009.

4. S. De Jong, "Simpls: an alternative approach to partial least squares regression," *Chemometrics and intelligent laboratory systems*, vol. 18, no. 3, pp. 251–263, 1993. [Online]. Available: http://www.sciencedirect.com/science/article/pii/016974399385002X

5. H. Xia and M. Bakker, "Single-shot libs spectral quality for waste particles in open air," *tm-Technisches Messen*, vol. 82, no. 12, pp. 606–615, 2015. [Online]. Available: http://www.degruyter.com/view/j/teme.2015.82.issue-12/teme-2015-0042/teme-2015-0042.xml

6. C. Alonso, C. Andrade, and J. Gonzalez, "Relation between resistivity and corrosion rate of reinforcements in carbonated mortar made with several cement types," *Cement and concrete research*, vol. 18, no. 5, pp. 687–698, 1988. [Online]. Available: http://www.sciencedirect.com/science/article/pii/0008884688900919

7. *European Committee for Standardization, EN 12620+A1: Aggregates for Concrete*, Std., 2008.