

Effect of compaction and thermal de-coating pre-treatments on the recyclability of coated and uncoated aluminium

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Abstract

Scrap pre-treatments, such as compaction and thermal de-coating, are standard industrial practices for recycling aluminium post-consumer scrap. This study compares the recyclability of a coated and uncoated 8111 alloy under the application of compaction and/or thermal de-coating pre-treatments. Sheets of 600 µm thickness were shredded into chips and compacted by uniaxial pressure, moderate pressure torsion (MPT) or MPT at 450 °C (Hot MPT) into briquettes of 4 cm diameter. A subset of briquettes and loose chips was subsequently heat-treated for 1 hour at 550 °C, while the other set was left untreated. The effectiveness of the heat-treatment for the different compaction methods was examined by mass balance and the internal porosity of the briquettes by computed tomography. Re-melting the samples under molten salt-flux showed that the coalescence of the coated material significantly improves with the thermal de-coating pre-treatment, especially for the loose chips and briquettes compacted uniaxially. Lower coalescences were obtained for the de-coated MPT briquettes, as a result of an incomplete de-coating.

Introduction

Aluminium food packaging scrap is often coated and/or contaminated with organic materials, which decreases the quantity and quality of the aluminium recovered. Applying a thermal pre-treatment is a common industrial practice to prevent issues associated with the moisture and organic content of this scrap [1]. Another challenging aspect in the recycling of aluminium packaging is the oxidation losses. [2] Compacting thin scrap into bales or briquettes reduces its specific surface area, an important parameter [3] on its susceptibility to oxidation during re-melting. This is already being done for some scrap types, e.g. chips from machining [4], [5] and used beverage cans (UBCs), since it facilitates storage and transport and, in the case of the chips, prevents them from floating when charged into the furnace. However, the importance of the degree of compaction (bulk density) on the oxidation losses is not yet clear. In a previous study, Vallejo-Olivares et al. observed that for clean aluminium foils compacted to briquettes of varied densities, compaction significantly reduced the oxidation during heat-treatment, especially for the thinnest materials. [6] For the current study, the research question is if "too much" compaction would negatively affect the de-coating process and if so, what consequences would this have on scrap re-melting? De-coating pre-treatments are beneficial in many ways, such as lower dross generation and improved process control and melt cleanliness. [7] Furthermore, re-melting un-treated packaging scrap could be the source of safety hazards such as the formation of H₂S (g), PH₃ (g), H₂ (g), or CH₄ (g), which are toxic, explosive or combustible [8]. The EN 139 standard [9] describes an average re-melting yield of 71.5 % for coated packaging and of 86.1 % for de-coated scrap. However, to compare these values, one must first carefully read the definitions. The metal yield is the percentage of metal gained from the total mass of the scrap. In contrast, the metal recovery represents the percentage of metal gained from the metallic mass of the scrap. Another parameter often addressed in research is the coalescence efficiency. It describes the ability of the individual aluminium pieces to merge, which is critical for a successful re-melting operation without too much metal loss to small metal droplets dispersed in the salt. It is however difficult to directly infer coalescence results from laboratory experiments to industrial set-ups, which may consist of rotary furnaces, magnetic stirring, etc.

Many researchers have studied the thermal de-coating pre-treatment to define the optimal time, temperature and atmosphere that secure an efficient de-coating while minimising aluminium oxidation. Kvithyld et al. [10] stated that there is a fine line between too little de-coating, exactly enough, and too much de-coating leading to oxidation. According to their weight loss analysis of a polyester coating, the complete combustion occurs at 550 °C, and higher temperatures just oxidise the aluminium. Capuzzi et al. [11] heat-treated and re-melted coated and uncoated aluminium disks under different salt-flux compositions and temperatures. They concluded that disks thermally

de-coated at 600 °C reached equivalent coalescence to those uncoated, while the treatments at 400 and 500 °C were not as effective. Gökkelma [12] assessed the recyclability of used aluminium coffee capsules, one of the packaging examples with higher organics/metal ratio, and obtained higher metal yield and coalescence efficiencies for the capsules that had been thermally pre-treated at 500 °C. The work performed by Steglich [13]–[16] constitutes one of the few research lines that include bale density as a parameter for thermal pre-treatment and re-melting. The most recent study [16] covers the recycling of UBC bales with different densities, organic content, and pre-treatment conditions in a multi-chamber furnace process. It showed that lower bale densities promote the removal of organics during heat treatment and that this results in a lower dross formation during re-melting. This partly answers our previously placed question: for aluminium scrap containing organics, denser bales can lead to a less effective heat treatment and, therefore, more re-melting losses. The present study aims to cast some more light into this matter by evaluating the interaction between the compaction, thermal de-coating and re-melting processes of an AA8111 aluminium sheet with and without coating. It is a continuation of the previous publication on the compaction, oxidation and re-melting of clean aluminium foil. [6]

Experimental Materials and Procedure

Two coils of aluminium sheet alloy AA8111 with 600 µm gauge were provided by Speira Holmestrand. One was coated with a lacquer, and this material will be referred to as «coated», while the other will be referred to as «uncoated». Table 1 contains the chemical composition of the produced alloy. Information on the coating composition and thickness was not available, so it was measured with a portable XRF analyser and SEM-EDS.

Table 1. Thickness and composition (wt%) of the materials and coatings studied

Material	Thickness	Al	Fe	Si	Mg	Mn	Cu	Zn	Ti	S	C	Remain.
AA8111 alloy ¹	600 µm	98.371	0.865	0.587	0.046	0.037	0.036	0.007	0.005	-	-	<0.05
Dark side (XRF) ²		87.2±6.1	0.68	4.56	-	-	-	0.017	5.43	2.03	-	
Light side (XRF) ²		94.5±0.5	0.69	2.55	-	-	-	0.01	2.01	0.19	-	
Dark coating ³	25 µm	0.35	0.04	18.28	0.15	-	0.1	0.18	2.13		57.84	
Light coating ³	5 µm	1.41	0.15	7.18	0.1	-	0.1	0.18	2.07		64.77	

¹Coil composition provided by Speira Holmestrand ²Both sides of the coated material composition were analysed by a portable XRF SPECTRO xSORT. ³The coating composition was analysed by SEM-EDS Zeiss Ultra 55LE FEG-SEM.

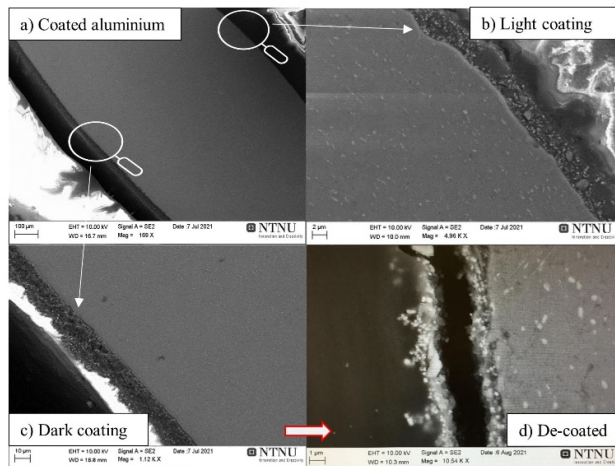


Fig. 1. SEM images a) Coated material. b) Higher magnification image of the light coating. c) Higher magnification image of the dark coating. d) Dark coating after thermal de-coating for 1 h at 550 °C.

One side of the coating was thinner (5 µm) and light in appearance, while the other was five times thicker (25 µm) and dark grey. As shown in Figure 1, the coating consists of a matrix and filler particles. An SEM-EDS point analysis taken in one of these particles revealed a composition of 21.26 wt% O, 20.75 wt% Ca, 12.30 wt% Ti, 4.76 wt% Si, and 23.81 wt% C. Although these analyses do not provide the exact composition of the coatings, they indicate that they consist of a polymeric matrix with filler particles of CaO, TiO and SiO₂. Figure 1d shows the dark coating after the heat treatment. The darker area shows a void (2 µm wide) surrounded by the oxide fillers that remained loosely adhered to the aluminium surface after the combustion of the organic

matrix.

Shredding and sieving

The coated and uncoated sheets were shredded into chips using a Getecha RS 1600-A1.1.1 with a grate of 8 mm diameter. Two sieves of square mesh 5 and 2 mm² were used to unify the size of the chips to this range, hence discarding 40-50 %wt of the material. Image analysis of 700 sieved fragments with the software ImageJ revealed an average area of 0.26 cm² and median area of 0.24 cm² for the uncoated chips and 0.26 cm² average and 0.23 cm² median for the coated chips, with a standard deviation of 0.14 cm² for both materials. The average weight per chip was estimated by dividing the weight of the chips analysed by their number. The mean weight was 49.0 mg for the uncoated and 48.4 mg for the coated chips.

Compaction into briquettes

The chips were compressed into cylindrical briquettes of 4 cm diameter, each weighing 20 grams, using a hydraulic press MTS 311. A subset was compacted by holding a 100 kN (80 MPa) uniaxial force during 5 s. This method will be referred to as uniaxial. Another subgroup was compacted by moderate pressure torsion (MPT), where the piston applied a uniaxial force of 70 kN (56 MPa) while the mould rotated 360 ° four times for 200 seconds. Finally, a third subset was compacted by moderated pressure torsion under 450 °C (Hot MPT). Half of the briquettes compacted uniaxially and by MPT were heat-treated. No heat-treatment was applied to the Hot MPT samples to evaluate whether the Hot MPT method would simultaneously serve as both de-coating and compaction. Figure 2 summarises the experimental procedure. The number of repetitions was 3 for each sample group, except for the Hot MPT with 2 repetitions. One sample from each subgroup was analysed by computed tomography (CT), giving a set of slices used to measure the internal porosity and reconstruct a 3D image.

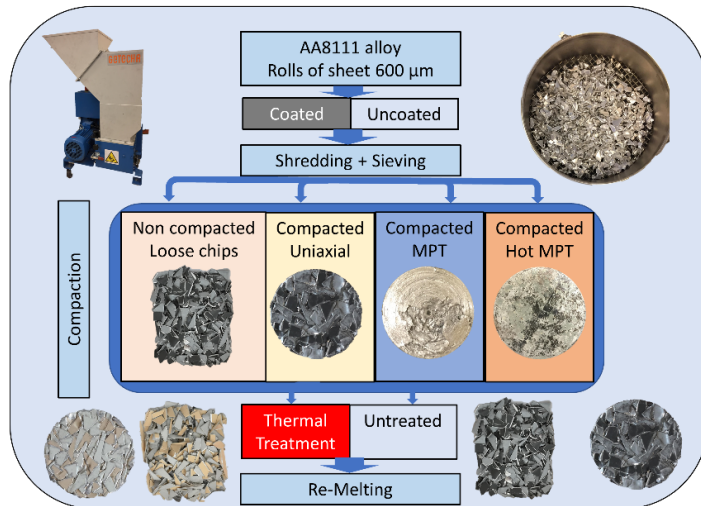


Fig. 2. Experimental procedure.

Thermal de-coating

The heat-treatment was performed in a Nabertherm Muffle Furnace with exhaust for 1 hour at 550 °C in air atmosphere. The samples were introduced into the furnace once the desired temperature was reached. A preliminary study was performed on sheets cut to 10x5 cm before selecting the above parameters, which also agrees with the values recommended in the literature [10, 11, 13]. Three temperatures were evaluated: 450 °C, 550 °C and 650 °C. Due to partial melting of the sheets at 650 °C and incomplete de-coatings at 450 °C, the temperature of 550 °C was confirmed. The treatment was also tested for 1h, 2h and 3h at this temperature. No significant improvements were observed for 2 h, and the weight increased after 3h due to oxidation.

Re-melting

The re-melting experiments were carried out in a Nabertherm resistance furnace. Three ceramic crucibles (Al_2O_3 -

SiO_2) of 20 cl volume, filled with 80 g of mixed salts with composition ratios (%wt) of 68.6:29.4:2.0 $NaCl:KCl:CaF_2$ were placed in the furnace at 800 °C. The aluminium samples were added into the crucibles once the salt was molten (after approx. 40 minutes). The crucibles were held in the closed furnace for 10 minutes at 800 °C, removed and naturally cooled in air. For the chips and briquettes of coated material that had not been thermally pre-treated, the spontaneous combustion of the coating generated flames and dark smoke for around 30 seconds. The furnace lid was kept open until the end of this combustion. Once the crucibles were at room temperature, the salt was separated from the metal by crushing and washing it with water on an 800 microns sieve, discarding any smaller particles. After drying, the metal pieces were weighted, and the metal yield, metal recovery and coalescence were calculated using Equations 1, 2 and 3.

$$\% \text{ Metal Yield} = \frac{m_{recov}}{m_{input}} * 100 \quad (1)$$

$$\% \text{ Metal Recovery} = \frac{m_{recov}}{m_{metal\ input}} * 100 \quad (2)$$

$$\% \text{ Coalesced} = \frac{m_{coalesced}}{m_{input}} * 100 \quad (3)$$

$$m_{metal\ input} = m_{input} - m_{input} * \% \text{ average weight loss} \quad (4)$$

Where m_{recov} is the sum of the masses of the pieces recovered, m_{input} is the mass of the briquette or batch of chips before re-melting, $m_{coalesced}$ is the mass of the biggest piece, and $m_{metal\ input}$ is calculated based on the average weight loss results for each compaction route: 1.66 % for loose chips, 1.70 % for uniaxial, and 1.52 % for MPT briquettes. This way, we can estimate the metal content of the sample before re-melting and calculate metal recovery values. It is assumed that after heat-treatment, the samples are 100 % metal, so for the uncoated and the de-coated samples, metal yield is equal to metal recovery.

Results and Discussion

Compaction

Table 2 presents the average bulk densities after briquetting by the different compaction methods and the briquette's internal porosity of both untreated and thermally treated briquettes. The internal porosity was measured by computed tomography (CT) for one sample from each group. The analysis was performed slice by slice using the software ImageJ, first adjusting a threshold to differentiate between material and void and then conducting a porosity measure. The values presented for each sample are the average of between 80-250 images and the std. Dev within sample ranged between 0-3 %. Slices close to the top and bottom of the briquettes showed higher porosity values, but they were omitted since they represent the external porosity.

Table 2. Average briquette bulk density (g/cm^3) and internal porosity (%) for different compaction routes.

	Uniaxial		MPT		Hot MPT	
	Porosity	Density	Porosity	Density	Porosity	Density
Uncoated	16.64	1.94 ± 0.01	0.68	2.46 ± 0.01	0.04	2.60 ± 0.02
Coated	14.68	1.90 ± 0.18	4.48	2.19 ± 0.09	0.07	2.54 ± 0.07
Uncoated Thrm.Treat.	18.28		1.39			
Coated Thrm. Treat.	15.89		4.75			

The bulk densities reached for the coated chips were lower than for the uncoated. This could be due to a lower adhesion of the coatings, higher thickness leading to stiffer chips, or both. Coated briquettes sometimes fell apart completely. In most cases, some loose chips fell off when taking them out of the mould and in further handling, especially for the briquettes compacted by the uniaxial method. The internal porosity drastically decreases when compacting by the MPT method, from the range 14.7-18.3 % to 0.7-5 %, and down to 0.04-0.07 for the Hot MPT method. The porosity is higher for the uncoated material than for the coated material compacted uniaxially but

higher for the coated material that had been compacted by MPT and Hot MPT. Both coated and uncoated materials present higher porosity after the thermal treatment, which could be attributed to the elimination of the coating and other volatiles or to a slight re-arrangement of the position of the chips due to thermal expansion during the treatment. These porosities are lower than those found in Steglich's [13] study on UBC bales (range 59-83 %), which correspond with today's industrial values.

During MPT of coated chips, some metal got stuck in the piston resulting in weight loss of the briquette and an uneven surface. Loose chips rarely fell off or got stuck for the uncoated briquettes, and the surfaces were smooth. These differences between the compaction methods are shown in the CT 3-D reconstructions in Figure 3.

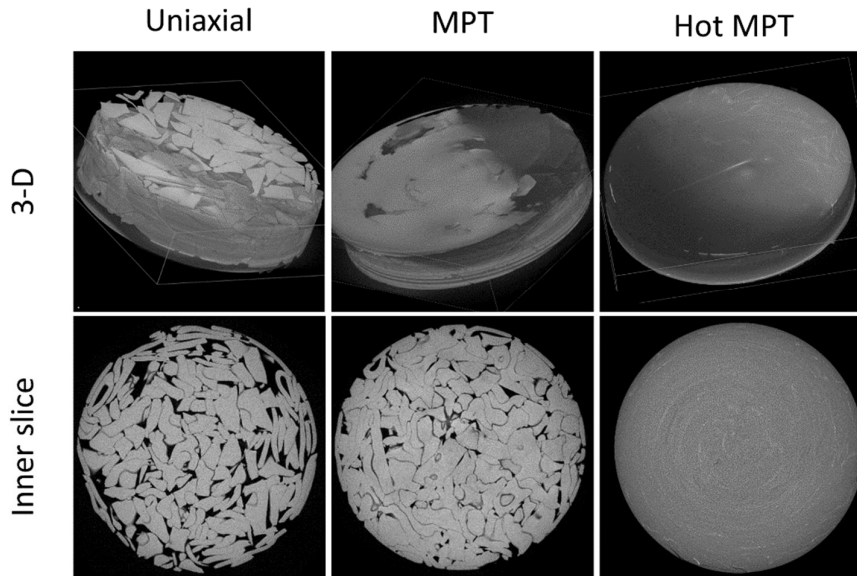


Fig. 3. Computed tomography of briquettes of coated material compacted by Uniaxial, MPT and Hot MPT method. **Top row:** 3D reconstruction. **Bottom row:** one of the inner planes captured showed the chips' internal porosity and deformation.

Thermal de-coating

The coated chips and briquettes experienced a colour change, shown in Figure 2, and became very fragile after thermal treatment. The uncoated briquettes showed no visible change and held together also after the treatment. Figure 4 shows the average weight change due to thermal treatment for coated and uncoated chips and briquettes.

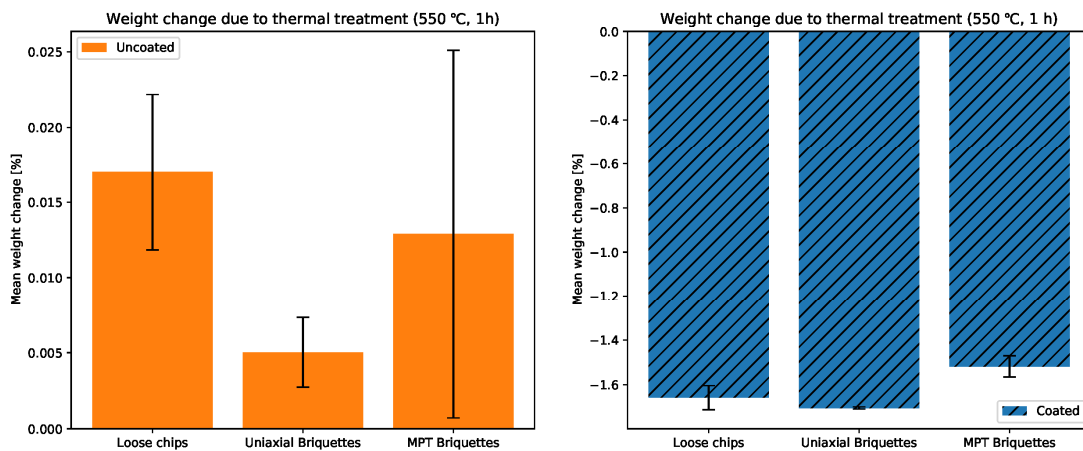


Fig. 4. Average weight changes due to thermal treatment for the uncoated (left) and coated (right) material. The bars show the std. Dev between the three repetitions. Note the difference in the y-axis scale between both graphs.

For the coated material, the uniaxially compacted briquettes lost, on average, almost the same weight as the loose chips: 1.70 vs 1.66 %. The coated MPT briquettes lost less weight on average: 1.52 %. This indicates that uniaxial compaction does not reduce the effectiveness of the thermal de-coating, in contrast to MPT compaction. All samples increased in weight for the uncoated briquettes due to oxidation, and the loose chips experienced the

highest average weight increase: 0.02 %. Still, the weight increases due to oxidation are two orders of magnitude lower than the weight decreases due to the de-coating. Therefore, the heat-treatment parameters (1h, 550 °C) seem to offer the pursued effects: effective coating removal and low oxidation. The oxidation results agree with some of the conclusions from the previous study [6], where briquettes were observed to oxidise less than loose chips. A lower oxidation for MPT than for uniaxial compression was expected, based on the lower internal porosity of the MPT briquettes. While the results indicate the opposite, the standard deviation in the data is large, making the result tentative. Finally, the fact that the uniaxial compaction, even up to densities of 2 g/cm³, does not affect the heat-treatment compared to the loose chips seems to oppose some of Steglich's observations, where bales compacted to lower densities generated higher dross, attributed to an incomplete de-coating. Crucial differences in their experimental method (re-melting without salt-flux) and material type (post-consumer scrap) may be the cause for the deviating observations.

Re-melting

Coalescence

The material recovered after re-melting showed various degrees of coalescence. Good coalescence leads to most of the chips merging into one round piece. On the contrary, poor coalescence leads to multiple small pearls distributed in the salt. A coalescence analysis can be done by comparing the recovered metal images in Figure 5a and the average coalescences for the different routes in Figure 5b. The results are collected in Table 3, in the following sub-section, together with the metal yield and metal recovery values.

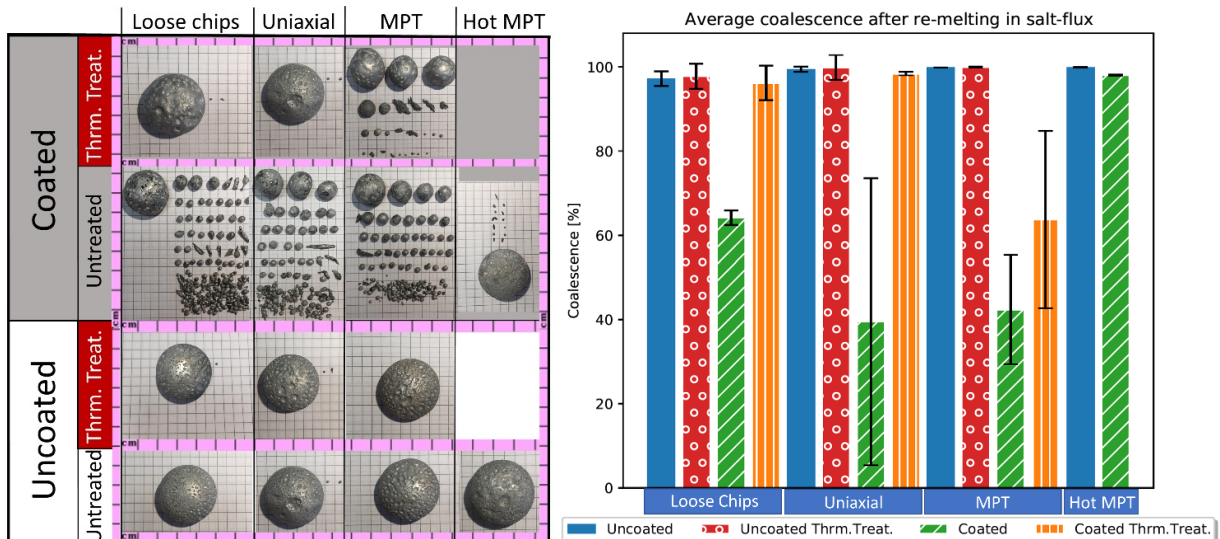


Fig. 5. Left: a) Example of the recovered metal for each of the compaction and thermal pre-treatment routes. Right: b) Coalescence results for all re-melting experiments. The bars show the standard deviation between repetitions.

The surfaces of the metal recovered from the coated, untreated samples appear rougher and darker because of the spontaneous combustion that took place during their re-melting. On the contrary, the re-melted uncoated and de-coated materials look smooth and shiny. The average coalescence results demonstrate (in agreement with literature [11, 12]) that applying a thermal de-coating pre-treatment has a great effect on improving the coated materials' coalescence. Regarding the compaction route, thermally de-coating either loose chips or uniaxial briquettes resulted in similar coalescences to those of the uncoated materials. However, the coalescence was lower for the de-coated MPT briquettes. The reason may be that the MPT briquettes are so tightly compacted that they inhibit part of the de-coating process, leaving some carbonaceous materials trapped inside the briquette and limiting the ability of chips to coalesce together, as previously observed by Steglich [20]. This is consistent with the results from the previous section. The uncoated material reached almost perfect coalescences regardless of the pre-treatment routes. However, a slight increase can be observed for the MPT and Hot MPT samples.

Metal yield and metal recovery

Table 3 collects the coalescence, metal yield and metal recovery results for all re-melting experiments. As mentioned in the introduction, these are different ways of reporting recycling efficiency. For comparisons between

samples with different non-metallic content, metal recovery values would be more accurate than metal yield. For most of the samples of this study, though, the recycling yield and recovery are the same. Therefore, some of the cells in the table are merged and present one value for both.

Table 3. Coalescence (C), Metal Yield (Y) and Metal Recovery (R) results.

	Loose chips			Loose chips Thrm. Treat.			Uniaxial			Uniaxial Thrm. Treat.			MPT			MPT Thrm. Treat.			Hot MPT		
	C	Y	R	C	Y	R	C	Y	R	C	Y	R	C	Y	R	C	Y	R	C	Y	R
Uncoated	95	95		94	94		100	100		103	103		100	100		100	100		100	100	
Uncoated	98	98		100	100		100	100		100	100		100	100		100	100		100	100	
Uncoated	99	99		100	100		99	99		96	96		100	100		100	100		100	100	
Average	97	97		98	98		99	100		100	100		100	100		100	100		100	100	
Std. Dev	1.7	1.6		3.0	3.0		0.6	0.6		3.0	3.0		0.0	0.0		0.1	0.1		0.0	0.0	
Coated	65	96	97	90	100		85	95	96	98	98		29	96	98	62	97	98	98	98	
Coated	62	94	96	99	99		3	96	98	99	99		60	96	98	91	95	98	98	98	
Coated	66	97	99	99	99		30	94	95	99	99		39	96	97	39	96				
Average	64	96	97	96	99		40	95	96	98	98		42	96	98	64	96			98	
Std. Dev	1.8	1.3	1.4	4.0	0.4		34.0	1.1	1.1	0.4	0.4		13.0	0.1	0.1	21.0	0.6			0.1	

The calculated metal recovery is just 1-2 % higher than the yield for the untreated coated samples. The difference would be more relevant when recycling materials with higher organics content, such as post-consumer scrap. Regarding coalescence, while the difference between coalescence and yield/recovery values is very small or non-existent for the uncoated and the de-coated samples, it can become as high as 90 % for the most extreme cases of coated untreated samples. Thus, in this study, coalescence is the most relevant parameter. If only the recovery or yield were evaluated, all the re-melting results would lay within the ranges 94-100%. This would not explain the differences between the products obtained via the different pre-treatment routes, shown in Figure 5a.

Conclusions

This work has investigated the compaction of coated and uncoated chips of aluminium sheet alloy AA8111 with 600 µm gauge by three different methods: uniaxial, moderate pressure torsion (MPT), and Hot MPT at 450 °C. The performance of thermal de-coating and re-melting in salt flux of these materials has been investigated. The following conclusions were drawn:

- The uncoated material was easier to compact than the coated, meaning more densely packed briquettes.
- The internal porosity of the briquettes drastically decreases when adding torsion to the compaction process and even further by adding temperature.
- A thermal de-coating of 1 h at 550 °C removed the organics while keeping the oxidation low; the weight of the coated samples decreased by 1.5-1.7 %, and it increased by less than 0.02 % for the uncoated.
- The coalescence of the coated samples improved due to the thermal de-coating: by 32 % for the loose chips, 58 % for uniaxially compacted briquettes, and 22 % for the MPT compacted briquettes.
- Re-melting de-coated chips and uniaxial briquettes resulted in similar coalescences to those of the uncoated materials.
- An incomplete de-coating is crucial for coalescence, as observed for the de-coated MPT briquettes; just a slightly lower de-coating weight loss (0.18 %) than the uniaxial but 35 % lower coalescence.
- Average metal yields lay between 95-100, and recoveries between 97-100 %.
- While it is possible to observe small differences between the yield and recovery results (averages below 98 % for the coated-non-heat-treated samples, uncoated loose chips, and de-coated MPT briquettes), the coalescence is more representative of the recycling efficiency in the present study.

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