



From automotive shredder residue to nano-ceramics and graphitic carbon—Thermal degradation kinetics



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ABSTRACT

Automotive shredder residue (ASR) has become a challenging problem due to its large and increasing production volumes. ASR consists primarily of plastics of high calorific value, making its processing by thermochemical processes a viable solution. The aim of this study is to develop a new sustainable approach, in which syngas, tars and valuable nano-ceramics can be generated from the fast and high-temperature pyrolysis of ASR. This work consists of two parts; in the first part, the characteristics and kinetics of ASR non-isothermal degradation were studied by correlating the thermogravimetric data via four models namely, Coats–Redfern model, Freeman and Carroll model, distributed activation energy model (DAEM) and Flynn–Wall–Ozawa model (FWO). Coats–Redfern model was not applicable to obtain the kinetic parameters of ASR pyrolysis, while the other models gave nearly similar and reasonable results. In the second part, ASR was treated by fast pyrolysis at 1550 °C and the carbonaceous residues were analysed. The carbonaceous residues were found to contain reasonable quantities of TiN and SiC nanoparticles that can be valuable precursors to produce hard ceramics for many applications. Other materials such as graphite and calcium magnesium aluminosilicate microspheres coated with TiN were also identified in the carbonaceous residues.

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1. Introduction

ASR waste is generated in the shredding facility of end-of-life vehicles (ELVs) where ELVs are dismantled and shredded into fist-sized chunks. Around 75–80 wt.% of shredded ELVs, mainly ferrous and non-ferrous materials, are separated by magnetic separation and electric conductivity-based separation (ECS) techniques. Ferrous and non-ferrous materials are recycled, while the remaining polymeric materials (20–25 wt.% of ELVs), for which recycling processes are not in place yet, are disposed of in landfills [76]. This fraction of materials is the ASR; it can be further upgraded to remove dust and other dirt through post-shredding technologies. Upgraded-ASR consists of plastics that are incorporated with reasonable content of TiO₂, which is initially added to the plastics in

automobiles to prevent their phototendering caused by the exposure to sunlight [23,32,8].

Although there is insufficient data about the global production of ASR, in 2002, [76] predicted it at around 10 million tonnes. However, it is believed that the global production nowadays is much greater due to the large and increasing trend in the vehicle ownership and also due to the modern design of automobiles which has shorter life span and lighter weight and involves more polymers [44]. In some countries, concern about the large volumes of ASR has emerged recently, and some regulations and directives regarding ELVs were introduced [61,1,2]. These directives, in summary, suggest more ASR to be recycled and impose restrictions on its disposal. This has led researchers to look for robust and economically viable solutions to recycle ASR. Several solutions were proposed such as pyrolysis, gasification, co-incineration with other waste streams and use as cement feedstock [26,56,31,59,69]. Among these processes, only pyrolysis seems to be the most promising as other processes suffer from several drawbacks. For example, co-incineration of ASR generates

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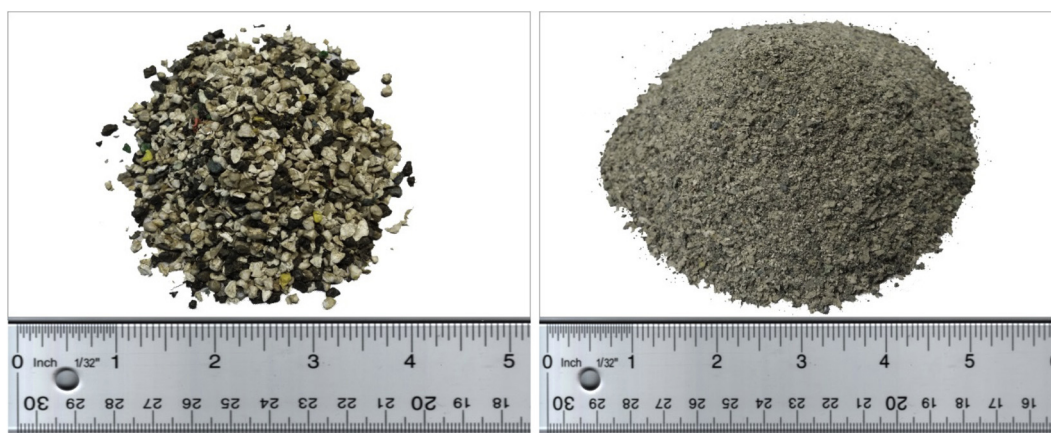


Fig. 1. As-received ASR (left side) and ASR powder (right side).

considerable amounts of harmful by-products such as NO_x , SO_x , POPs, dioxins and furans [71], while the use of ASR as a cement feedstock results in poor quality clinker with undesirable content that might limit its use [31]. In contrast, pyrolysis does not involve air or oxygen, markedly limiting some reactions that generate deleterious emissions such as NO_x , SO_x , dioxins and furans. Pyrolysis also can generate useful by-products including pyro-gas (i.e., CH_4 and Syngas), tars and chars. Although several studies investigated the pyrolysis of ASR, the recovery of energy from ASR by pyrolysis remains commercially and technically unproven and needs further investigations. In addition, previous studies only investigated the pyrolysis of ASR at moderate temperatures between 450 and 800 °C [14,64,21,22,30,60,48,55,77]. Plastics of ASR have high thermal stability and their pyrolysis at moderate temperature requires prolonged heating, resulting in significant energy consumption. Furthermore, previous studies completely ignored the essential Ti content of ASR.

Titanium is widely used to produce ceramics with outstanding physical and mechanical properties such as TiN, TiC and TiO_xNy . These ceramics are extensively used as hard and wear-resistant coatings for cutting-tool materials. They also can be used in micro-electronic devices and other industrial and military applications. Most of bulk ceramics are conventionally fabricated in electrical furnaces from a mixture of naturally-occurring mineral oxide (i.e., TiO_2) and petroleum coke via carbothermic reduction and nitriding reactions at high temperatures [24,58,72]. The resulting product is typically a mixture of 10–30% bulk ceramics (large particle size $\sim 1 \mu\text{m}$), 15–30% free carbon, 40–60% unreacted mineral oxides and 1–5% ash. Ceramics can be then separated from this mixture and purified by mature technologies [34]. In this process, the morphology and particle size of the resulting ceramics are difficult to control. Large particle size ceramics (i.e., monoliths) usually have insufficient mechanical strength and are difficult to process [33,54]. For this reason, nano-ceramics and their fabrications techniques have recently received great attention. Several techniques have

been developed to fabricate nano-crystalline TiN including reactive ball mill method (RBM) [65], direct nitriding [7], sol-gel [41,42] and plasma chemical synthesis [62]. In the RBM method, pure Ti nano-powder is milled using a high energy ball mill at room temperature under nitrogen flow for prolonged periods of time. The resulting nanoparticles have a very small particle size around 5 nm. Such small particles are difficult to be further processed by the conventional sintering techniques since small nanoparticles are highly reactive and have high tendency to oxidize. In the direct nitriding method, TiO_2 nano-powder is simply purged with ammonia at temperatures between 700 and 1000 °C. However, the morphology and particle size are difficult to control and greatly depend on the structure of the TiO_2 precursor. Sol-gel and plasma chemical synthetic techniques require many different chemicals; some of these chemicals can pose threat to humans and the environment.

In the present study, we suggest a sustainable solution for ASR processing where TiN nanoparticles with ideal particle size ranging from 200 to 315 nm can be formed as a by-product. In this process, fast pyrolysis of ASR at 1550 °C is adopted. At this temperature, the Ti, carbon and nitrogen contents of ASR undergo carbothermic and nitriding reactions, resulting in reasonable quantities of TiN and SiC nano-ceramics along with graphitic carbon and small amounts of alumina and lime. Graphitic carbon can be simply removed by ashing the carbonaceous residues in an air-open furnace at 650 °C [28], while the small amount of alumina and lime can be very useful in the subsequent sintering process as the presence of these oxide can contribute to the physical and mechanical characteristics of the resulting ceramics [29,11,39,45,63]. Although the process involves high temperature, the energy consumption is not unreasonable as the decomposition of ASR at high temperatures can be completed within a short time. In addition, valuable by-products such as TiN nanoparticles can be synthesized from only waste materials without using conventional resources or synthetic chemicals.

Table 1
Composition of ASR.

Proximate analysis		Ultimate analysis					
Polypropylene	30–39%	C (%)	7.3–13.8	Br (%)	1.5–2.4	I (ppm)	52.9
Polyethylene	21–28%	Ti (%)	1.8–3.2	Cl (%)	0.19–0.23	K (ppm)	826
Polyurethane	9–14%	Ca (%)	0.8–1.1	N (%)	0.3–0.38	Mn (ppm)	18.4
Polycarbonate	7–12%	Si (%)	0.5–0.9	Ba (ppm)	102.7	Na (ppm)	325.1
Textiles	1–2%	Mg (%)	0.2–0.3	Cr (ppm)	35.0	Ni (ppm)	12.6
Moisture	0.8–1.1%	S (%)	0.3–0.4	Cu (ppm)	27.2	P (ppm)	583
Ash	2–3.9%	Al (%)	0.1–0.12	Fe (ppm)	88.7	Pb (ppm)	45.5
Others	4–8%	Sb (%)	1.5–2.3	Hg (ppm)	14.7	Sn (ppm)	361

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