Preparation and Analysis of Plastic Reference Materials for Elemental Analysis

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Introduction

Because the reference materials(RM) and the dissolution procedure of plastic sample have not been yet established. The analysis of trace elements in plastic materials has known to be a very difficult technique1-3. Recently, the necessity of elemental analysis in plastic products has significantly increased in the environmental and material sciences3-6. In EMC(epoxy molding compound) for example α-emitters and ionic impurities such as U, Th and Na, Cl, Fe must be analyzed to very low level4.

In the present work, preparation and analysis of plastic RM have been studied. Plastic reference materials were prepared by mixing polypropylene powder and selected organometallic compounds which were commonly used in plastic industry as functional additives. Polypropylene powder and additives were mixed in a rotary twisting mixer with optimum mixing condition. For the progress homogeneity and stability, the mixture of powder was extruded to pellets about 2mm diameter with a thermal extruder. The pellets were distributed in several sample bottles with a sample divider. Additives were Al, Ca, Mg and Zn which were organometallic compounds. The homogeneity of metallic elements was tested by determining the concentration of elements after random sampling by the use of NAA. Other analysis methods of ICP-AES, AAS, and XRF which were commonly used to analysis of metallic elements in various materials were also studied and discussed.

Experimental

Plastic raw material and additives were collected by the investigation of physical properties, mixing characteristics and stability. The concentration and homogeneity of inorganic elements in the collected materials were investigated by NAA. A polypropylene(PP) powder and four additives of Al, Ca, Mg and Zn were selected in this work. To improve the homogeneity of the powder mixture of reference materials, both hand and instrumental mixing were performed. The mixture was made to pellets with a thermal extruder. Four kinds of pellets with different element concentration were made by this mixing and extruding processes. A sample divider was used to distribute

pellets homogeneously in several sample bottles. Concentration of elements in the pellets were investigated by NAA, ICP-AES, AAS and XRF. While the NAA method was established in the previous work1,2, other analytical methods were not be established. The absolute concentration of each element obtained from other analytical methods could not be trusted, however the relative standard deviations(RSD) of repeated analysis by each method could be used to evaluate the homogeneity.

Result and Discussion

Elemental concentration and homogeneity of starting materials such as PP powder and additive chemicals are very important, because the quality of reference materials should be affected directly by them. Table 1 shows the concentration and homogeneity of trace elements in PP powder by NAA. The concentration of Al, Ca, Mg, Zn in the selected PP powder were lower than tens ppm. And the homogeneity of the elements were not too bad in principle. Because the concentration of the elements in plastic RM was decided higher than tens ppm, the concentration of elements in PP powder would be negligible. The analytical result of PP pellets by NAA was presented in Table 2. The results by other methods were presented in Table 3. All elements in plastic RMs were distributed lower RSD values in NAA results, while the RSD values from others were higher than 10%. This high RSD values were not clear whether the homogeneity of RMs or analytical uncertainty. However, considering of NAA and XRF are nondestructive techniques, the high RSD values may not come from homogeneity, but from analytical uncertainty. We are going to confirm the source of high RSD within near future using various nondestructive and destructive analysis methods.

References

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Table 1. Concentration and homogeneity of metallic elements in the raw polypropylene powder selected for plastic reference materials

(ppm)

Element	1	2	3	4	5	Mean ± SD
Al	12.2	11.7	12.1	11.9	12.0	12.0 ± 0.2
Ca	0.350	0.370	0.360	0.370	0.360	0.362 ± 0.008
Mg	2.23	2.03	2.15	2.18	2.12	2.14 ± 0.08
Na	0.230	0.220	0.220	0.240	0.250	0.232 ± 0.13
Cr	0.039	0.037	0.038	0.037	0.039	0.038 ± 0.001
Cu	0.056	0.063	0.064	0.052	0.057	0.058 ± 0.005
Fe	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
K	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Mn	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Co	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
V	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
As	<0.1	<0.1	<0.1	<0.1	<0.1	<0.1
Cd	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Zn	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01

Table 2. Analytical results of plastic reference materials by NAA

(ppm)

SRM	value	Al	Ca	Mg	Zn
PP-1	mean	5810	8210	8540	9750
	RSD	1.7	1.68	1.01	2.32
PP-2	mean	650	931	934	945
	RSD	3.17	2.78	2.21	2.35
PP-3	mean	103	104	98.2	102
	RSD	1.63	2.86	1.51	2.07
PP-4	mean	43.9	11.2	17.1	12
	RSD	1.0	2.16	3.24	2.3

Table 3. Analytical results of plastic reference materials by other methods

(ppm)

SRM	Method	value	Al	Ca	Mg	Zn
PP-1	ICP	mean	4600	7040	6380	7760
		RSD	4.35	0.78	11.1	1.73
	XRF	mean	3896	4931	-	5468
		RSD	1.11	1.04	-	1.64
	AAS	1ea	4600	3011	5631	507
PP-2	ICP	mean	534	768	1017	828
		RSD	2.23	1.89	2.2	1.32
	XRF	mean	561	811	-	1010
		RSD	1.32	0.08	-	0.6
	AAS	1ea	366	648	994	29
PP-3	ICP	mean	87.4	85	116	88.8
		RSD	3.09	1.18	4.04	3.42
	XRF	mean	128.1	94.6	-	113
		RSD	4.88	1.19	-	1.23
	AAS	mean	105	78.3	118	45
		RSD	3.84	11.4	5.93	53.9
PP-4	ICP	mean	36	9.38	16.4	9.68
		RSD	2.78	1.01	3.34	2.39
	XRF	mean	63.9	9.82	-	6.69
		RSD	7.43	3.71	-	13.9
	AAS	mean	48.7	9	14	6.33
		RSD	7.78	11.1	12.4	9.12

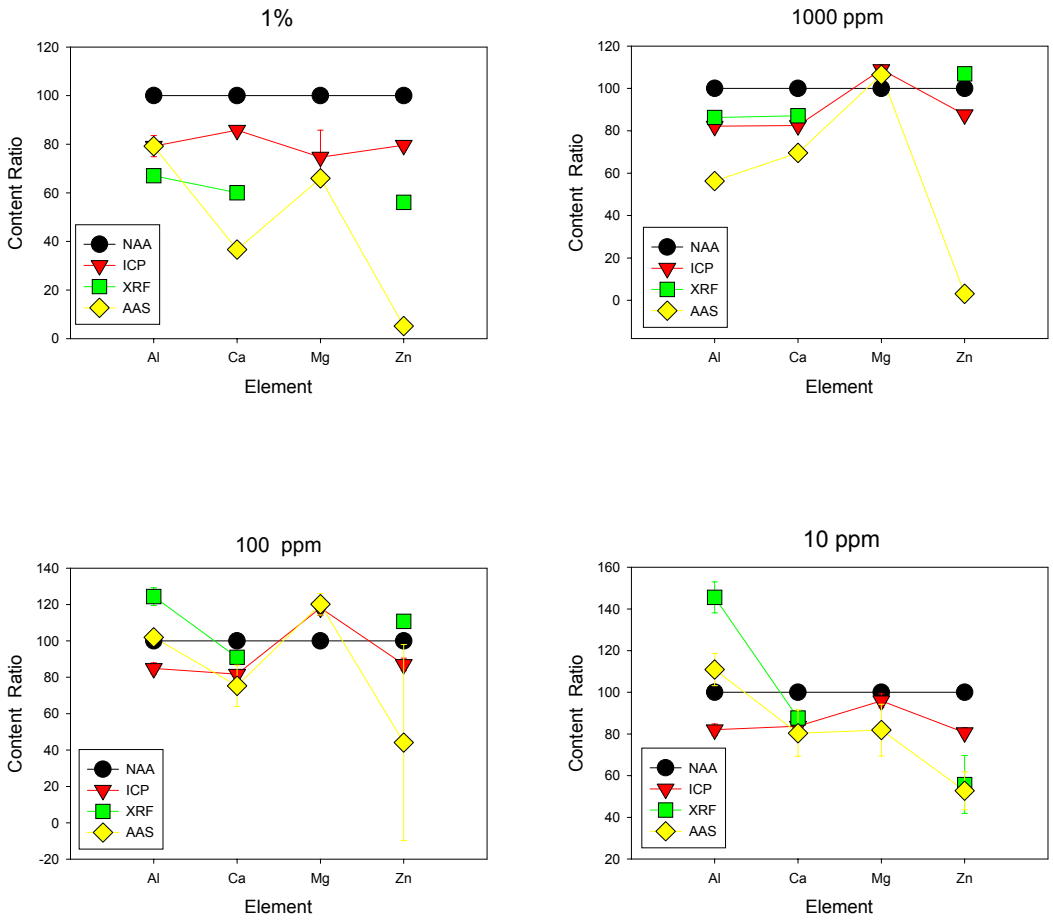

Fig. 1. The content ratios based on NAA and RSD values with analysis methods.