

substantiated the suitability of fitted response models and satisfactorily predicted the variation in processing parameters. The optimum process conditions for preparation of novel aonla candy were SSC 60°B, TD 30 min and DT 70°C. The study confirms the feasibility of development of this innovative product with a greater nutritive value, which also may attract more attention in the market.

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Potential application of yellow gypsum from LD slag as a soil conditioner

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In the present study, ICP-AES and ICP-MS techniques were used to analyse and validate the content of heavy and trace elements in yellow gypsum samples produced by a chemical process from a solid waste generated at an integrated steel plant. The present study tries to demonstrate that the content of heavy metals like lead, mercury and arsenic in yellow gypsum is less than the toxicity characteristic leaching procedure (TCLP) norms set for a comparable industrial by-product phospho-gypsum. This study also concludes that yellow gypsum contains some elements (copper, boron, etc.) in traces, apart from heavy metals which are beneficial to plants as micronutrients.

Keywords: Heavy metals, ICP-MS, ICP-AES, trace elements, yellow gypsum.

QUANTIFICATION of hazardous, heavy and trace elements in products, particularly produced from industrial wastes is very important for their possible applications in various sectors especially in agriculture. Efforts are being made worldwide by scientists for economic utilization of steel plant wastes. One such effort is the production of high purity iron and CaO-based sorbent using acetic acid by Tian *et al.*¹ and secondly production of yellow gypsum and gypsum by Ashrit *et al.*^{2,3}

Yellow gypsum produced from the Ling-Donawitz (LD) slag fines has very good plant nutrient value in terms of calcium, iron, phosphorus, magnesium and silicon, apart from various other trace elements beneficial for plants like boron, copper, manganese, etc. Environment protection agency (EPA), USA has also studied the quantity of contaminants mainly heavy metals and non-nutrient metals present in various fertilizers as a part of information gathering initiative⁴. Such studies are essential as these contaminants enter the food chain through plants and vegetables. To quantify the heavy and trace elements present in yellow gypsum, sample preparation is an important step of the analysis. Lithium metaborate fusion dissolution method and open acid digestion with different acid combinations followed by inductively coupled plasma-mass spectroscopy (ICP-MS) analysis were discussed by Roy *et al.*⁵. Similarly, Cremer and Schlocker⁶ had discussed about lithium borate fusion

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technique for dissolution of zircon and fluorite. Lithium metaborate dissolution for rocks, minerals and ores was originally proposed by Ingamells⁷, and along with Suhr⁸ later suggested its use for analysis via flame atomic absorption spectrophotometry technique. The major disadvantage of this type of dissolution is that the silica concentration should not exceed 150 parts per million, otherwise it will result in the polymerization and precipitation of silica. On the other hand if the solution is too dilute, low levels of constituents cannot be determined accurately as described by Shapiro and Brannock⁹. Thus, the digestion of silicate samples often remain unfinished^{10,11} and affects the accuracy and precision of elements to be estimated. The most commonly used methods of sample preparation either employ dissolution by fusion or acid digestion involving mixtures of acids such as HF + HNO₃, HNO₃ + HCl, HClO₄ + HF, etc.

Inductively coupled plasma atomic emission spectrometry (ICP-AES) is accepted widely and extensively used for estimation of minor and trace elements present in the ores, minerals and other related samples^{12,13}. ICP-MS has been used as an analytical technique for analysing the trace elements in geological samples^{14,15}, however, we have used it now for the analysis of synthetic gypsum produced from chemical treatment of steel plant waste.

ICP-AES is also a well established technique in the field of estimation of trace which was done by neutron activation analysis (NAA) since few decades, and estimation of major elements was by X-ray fluorescence spectrometer (XRF). However, ICP-AES is gaining momentum and gradually becoming more popular, as major and trace elements can be determined together using ICP-MS^{16,17}. ICP-MS is recognized as an effective technique for the determination of trace elements including rare earth elements in ceramics, etc.¹⁸. Thus both the methods have been well established to be the dominant methods for trace level detection of elements.

It is now apparent that combination of ICP-AES and ICP-MS techniques has huge potential for the determination of large chunks of trace elements and rare earth elements in various minerals, ores and geological samples. A major difficulty to overcome in the process of estimation of trace elements is the sample dissolution step, where the use of appropriate alkali or acid to achieve complete dissolution is critical.

There are several methods of sample dissolution techniques used by various researchers¹⁹⁻²¹; these include partial digestion (acid leaching) and fusion with alkali fluxes including certain methods developed for specific applications (mainly for determination of one or few elements). Alkaline fusion with NaOH + Na₂O₂ was also followed for determination of rare earth and other trace elements by ICP-MS²². Sodium carbonate fusion is a common procedure used mainly for silicates (clays, minerals, glass, rocks, slags, etc.) refractory oxides, insoluble phosphates and sulphates²³. Several acids are com-

monly used in wet ashing procedure. The dissolution process in this method is simply a replacement of hydrogen by metal²⁴. The main objective of the present work was to explore the potential of yellow gypsum as a synthetic soil conditioner produced from wastes generated in steel making with the help of ICP-AES and ICP-MS techniques. Also, the present study endeavours to demonstrate that the content of heavy metals like lead, mercury and arsenic in yellow gypsum is less than the toxicity characteristic leaching procedure (TCLP) norms set for a similar industrial by-product phospho-gypsum.

In the present study, a simple technique of sample decomposition was selected as compared to the one suggested by Yokoyama *et al.*²⁵ and Sharma *et al.*²⁶, wherein a combination of perchloric acid and HF was used. We have instead used aqua-regia as a reagent for sample dissolution, wherein 1 g of accurately weighed yellow gypsum sample was taken into a 250 ml teflon beaker and 40 ml of aqua-regia was added. The beaker was heated on a digital hot plate under a fume hood at controlled temperature for a time period so that the volume of the solution reduced to half, i.e. 20 ml. The aqua-regia solution was prepared by using concentrated

Table 1. Optimized inductively coupled plasma atomic emission spectrometry (ICP-AES) instrument operating parameters

Instrument parameters	Values
Model	Spectro, Arcos, Germany
Plasma power	1400 W
Pump speed	30 rpm
Coolant flow	14 l/min
Auxiliary flow	1.0 l/min
Acquisition time	45 sec
Number of replicates	3
Curve type	Linear

Table 2. Optimized ICP-MS instrument operating parameters

Instrument operating parameters	Values
Instrument	
Model	Perkin-Elmer, DRC-e
RF Power	1350 W
Plasma gas flow	1.5 l/min
Auxiliary gas flow	1.2 l/min
Nebuliser gas flow	0.88 l/min
Acquisition	
Scanning mode	Peak hopping
AMU	50 ms
Integration mode/time	Cps/1000 ms
Detector dead time	70 ns
Acquisition time	20 sec
Number of replicates	3
Curve type	Linear

Table 3. Comparing heavy metal concentrations with TCLP norms for phospho-gypsum

Trace and heavy metals in yellow gypsum	Values (mg/l)	TCLP test norms for phospho-gypsum (not to exceed mg/l)	Meeting the norms (yes/no)
Lead	1.7	5	Yes
Arsenic	0.08	5	Yes
Mercury	0.06	0.2	Yes

Table 4. Trace elements' concentration in yellow gypsum by ICP-AES and ICP-MS instruments

Parameters	Method	Values ($\mu\text{g/m}$)			Average ($\mu\text{g/g}$)	Standard deviation
		Repeat 1	Repeat 2	Repeat 3		
V	ICP-AES	43.67	41.01	40.85	41.84	1.58
Sr	ICP-AES	69.55	65.79	65.91	67.08	2.14
Sn	ICP-AES	42.23	40.63	40.37	41.08	1.01
B	ICP-AES	50.35	47.30	47.46	48.37	1.72
Cr	ICP-AES	115.8	117.4	118.5	117.2	1.36
Mo	ICP-MS	0.31	0.32	0.28	0.30	0.02
Ba	ICP-MS	11.56	10.94	10.87	11.12	0.38
Ni	ICP-MS	2.90	2.83	2.83	2.85	0.04
Co	ICP-MS	1.28	1.25	1.21	1.25	0.03
Cd	ICP-MS	1.31	1.29	1.28	1.29	0.02
Cu	ICP-MS	4.61	4.31	4.32	4.41	0.17

supra pure HCl and HNO₃. Water used in the sample preparation process was deionized water with a resistivity of >18.3 M Ω cm⁻¹ using a Milli-Q system. The reduced volume of solution was cooled, filtered and transferred quantitatively to a 100 ml volumetric flask.

The analytical characteristics of both the techniques, i.e. ICP-AES and ICP-MS setups used in the present work are described in detail, based on the study by Hein *et al.*²⁷. ICP-MS has been successfully applied for rapid and precise measurement of wide range of elements²⁸. However, heavy metal contents still cannot be easily measured due to difficulties in sample preparation, sensitivity limits of instrument and complicated matrix effects. The recent advances in ICP-MS technology like micro sample introduction methods, etc. have significantly improved the performance in quantification of trace elements.

ICP-AES determinations were carried out using Spectro ARCOS, spectrometer (Germany), with a working plasma power of 1400 W and other operating parameters as mentioned in Table 1.

The calibration of the instrument was performed using a blank prepared by using the acids in the same proportion, as used for sample preparation. Four standard multi-element solutions were prepared by diluting a concentrated multi-element standard solution containing all the elements to be determined by ICP-AES. For preparing the multi-element solution, National Institute of Standards and Technology (NIST) traceable multi-element E Merck standard solutions of 1000 ppm concentration were used (E Merck CRM 1V).

The ICP-MS determinations were carried out using Perkin Elmer DRC-e Instrument whose operating parameters are provided in Table 2.

Adoption of a suitable sample dissolution method is very important for the assessment of heavy metals and other elements present in traces. Several researches have examined dissolution process by using aqua-regia and total dissolution by using hydrofluoric acid to establish total concentrations²⁹. Other dissolution techniques like the application of HF for digestion with secondary acids mainly HNO₃, H₂SO₄ or HClO₄ were also studied¹⁸. Dissolution by alkali fusion involving either sodium carbonate or sodium hydroxide, where the determination of sodium is not required and finally the use of lithium metaborate as a flux useful for the determination of all elements including silicon in silicate dominant samples or many minerals containing silicon. The main drawback of these alkali fusion techniques is high flux to sample ratios like 5 : 2 and 7 : 2 at which the total dissolved solids in the resultant solution will be high and may end up with the choking of nebulizer. The method of dissolution described above has no such drawbacks and can produce recoveries more than 95% for certified reference materials (CRMs) used for validation of the method.

Table 3 provides the comparison of heavy and hazardous elements concentration in the yellow gypsum tested by ICP-MS, with the TCLP norms set for phospho-gypsum for application in agriculture as a soil conditioner. As the data (Table 3) indicates, yellow gypsum meets the norms set by TCLP tests and the concentration of heavy and hazardous elements in the yellow gypsum are

Table 5. Validation of results using multi-element standard solution using ICP-AES and ICP-MS instruments

Merck CRM IV	Method	Certified values (undiluted stock solution; mg/l)	Working values (working solutions; mg/l)	Observed values (mg/l)	Recovery (%)
Ba	ICP-AES	1000	100	99	99
Ni	ICP-AES	1006	100	102	102
Co	ICP-AES	1005	100	103	103
Cd	ICP-AES	1004	100	98	98
Cu	ICP-AES	1005	100	102	102
Sr	ICP-AES	1004	100	97	97
B	ICP-AES	1000	100	99	99
Cr	ICP-AES	1004	100	103	103
Ba	ICP-MS	1000	100	95	95
Ni	ICP-MS	1006	100	97	97
Co	ICP-MS	1005	100	101	101
Cd	ICP-MS	1004	100	99	99
Cu	ICP-MS	1005	100	103	103
Sr	ICP-MS	1004	100	97	97
B	ICP-MS	1000	100	98	98
Cr	ICP-MS	1004	100	101	101

Table 6. Validation of results by certified reference materials using ICP-AES and ICP-MS instruments

	Method	Certified values ($\mu\text{g/g}$)	Observed values ($\mu\text{g/g}$)	Recovery (%)
CRM no. SARM-5				
Ni	ICP-AES	555	545	98
V	ICP-AES	230	222	97
CRM No. 683-1				
Cr	ICP-AES	180	190	106
CRM No 681-1				
Pb*	ICP-MS	72	69	96
Co*	ICP-MS	80	85	106
As*	ICP-MS	139	135	97

*Reference values.

much lower compared to the TCLP values of leachate of phospho-gypsum^{30,31}.

Other trace elements present in the phospho-gypsum are provided in Table 4 and the data shows that the values for each individual element are much below the norms set by the government/regulatory authorities. Some elements like boron, vanadium, tin, copper, etc. are well recognized micro-nutrients for plants.

The results obtained by ICP-AES and ICP-MS methods were validated by using the CRM's and synthetic standard solutions traceable to NIST (E. Merck CRM IV), prepared under similar conditions along with the yellow gypsum sample.

E. Merck CRM IV was used for validation of elements concentration (Table 5), by adding the known quantity of major matrix elements present in the yellow gypsum to the synthetic standard and then recovery of the minor elements were calculated to establish the correctness of findings. E. Merck CRM IV solution was analysed both in ICP-AES and ICP-MS instruments. In a similar way, Bureau of Analysed Samples (BAS) Ltd standards like SARM-5 and 683-1 containing the elements of our inter-

est were prepared and the solution matrix was matched to yellow gypsum using the analytical grade calcium sulphate and the recovery was calculated as presented in Table 6 (analysed in ICP-AES and ICP-MS instruments).

Due care was taken in preparing the calibration curve covering the analysis range and linearity. The line of best fit with Y intercept close to zero and correlation coefficient of more than 0.9999 was used. Validation parameters of other methods like repeatability and ruggedness and intermediate precision were also carried out by analysing the sample several times and on different days.

It is evident from the present study that yellow gypsum is suitable as a soil conditioner as it contains better nutritional value in terms of sulphur, calcium, iron, phosphorus and silica with reference to mineral gypsum. Thus, yellow gypsum is important in the conservation of natural resources which helps in the effective and economical utilization of industrial solid wastes. It also contains traces of micro-nutrients like copper, boron, nickel molybdenum, etc. which are essential part of plant nutrition system. Silica, another useful constituent in yellow

gypsum, helps in plant growth and provides strength to stem and yield according to recent studies. Content of heavy metals and other hazardous metals is quite below the limit set by TCLP for the product of similar nature such as phospho-gypsum. Major advantage of yellow gypsum is its composition which is full of micro nutrients and hence, is fit for use as a soil conditioner.

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