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Characterization of Buffing Dust using Nigerian Research Reactor 1 (NIRR-1) and its Environmental Impact

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ABSTRACT

Buffing dust from chrome tanned leather which is proteinous, impregnated with Chromium, synthetic fat, oil, tanning agents and dye chemicals is one of the difficult tannery wastes to manage. Its Characterization using neutron activation analysis technique indicates that Al, Cr, Fe, K and Na contribute as much as 17,600ppm, 5,000ppm, 43,000ppm, 5,500ppm and 12,200ppm respectively, while As, Ba, Br, Ca, Co, Cs, Dy, Eu, Hf, La, Lu, Mg, Mn, Rb, Sb, Sc, Sm, Ta, Th, Ti, U, V, Yb and Zn contribute $3.5 \pm ppm$, $64 \pm 4ppm$, $3.1 \pm 0.02ppm$, $1.0 \pm 0.1ppm$, $3.4 \pm 0.2ppm$, $0.23 \pm 0.05ppm$, <0.7ppm, $0.06 \pm 0.01ppm$, <1.1ppm, $11.5 \pm 0.03ppm$, $0.03 \pm 0.005ppm$, <7250ppm, $153 \pm 21ppm$, $24 \pm 2ppm$, $0.8 \pm 0.05ppm$, $2.02 \pm 0.06ppm$, $2.1 \pm 0.1ppm$, <1ppm, <1.2ppm, <2500ppm, <1.5ppm, <15ppm, <0.9ppm and $769 \pm 21ppm$ respectively. The results obtained were validated with the data obtained from the simultaneous analysis of IAE-336 (Lichen) and IAEA 358 (Cabbage) which are all Internationally certified biological reference materials.

Keywords: Hides and skins; Buffing dust; Neutron activation; Radio nuclide; Reactor.

INTRODUCTION

The principal aim of the leather industry, which plays a significant role in today's economy, is to transform animal hides/skins into a physically and chemically stable material by subjecting them to chemical and mechanical sequential processes, and therefore to obtain products for meeting various needs of people. The leather industry uses hides and skins as raw materials, which are by-products of meat and the meat industry. In this respect, the leather industry could have been easily distinguished as an environmental friendly industry, since it processes waste products from meat production [1]. However, the leather industry has commonly been associated

with high pollution because of generation of huge amount of liquid organic wastes and high water consumption during manufacturing processes [2]; and solid wastes plus emission of obnoxious smell due to the degradation of proteinous material of hides and skins and generation of gases such as NH_3 , H_2S and CO_2 . Different forms of waste in quantity and quality, which emerge during transformation of hides and skins to leather have negative impacts on the environment.

Leather industry in the developing countries is facing lots of solid wastes problem and many tanneries closed for not meeting bio-chemical oxygen (BOD) demand and total solids (TDS) norms [3]. The tannery effluents are characterized by high contents of dissolved, suspended organic and inorganic solids giving rise to high oxygen demand and potentially toxic metal salts and chromium metal ion. Solid wastes generated in leather industries contribute mainly skin trimmings, keratin wastes, fleshing wastes, chrome shaving wastes and buffing wastes. It constitutes protein as the main component.

Out of 1000 kg of raw hide, nearly 850 kg is generated as solid wastes in leather processing. Only 150 kg of the raw material is converted into leather. Tannery generates huge amount of solid waste as follows: fleshing, 50-60; chrome shaving, chrome splits and buffing dust, 35-40; skin trimmings, 5-7; and hair, 2-5%. Solid wastes in leather processing constitute: beam house, 80; tanning, 19; finishing, 1% [3].

Neutron activation analysis (NAA) is a technique which, in its instrumental form, makes it possible to determine large number of elements in different types of matrices [4]. The Nigerian Research Reactor 1 (NIRR-1) installed and commissioned at the Centre for Energy Research and Training (CERT), Ahmadu Bello University, Zaria, Nigeria for training and Research was used for the analysis. The NIRR-1 is a Miniature Neutron Source Reactor (MNSR) and has a tank in pool structural configuration and a nominal thermal power rating of 31 (kW) with a built in clean cold core excess reactivity of 3.77 mk measured during the on-site zero power and criticality experiments. The reactor can operate for maximum of 4.5 hours at full power (i.e. equivalent to a thermal neutron flux of 1×10^{12} ncm⁻² S⁻¹ in the inner irradiation Channels). Under these conditions the reactor can operate with the same fuel loading for over ten (10) years with a burn up of <1% [5]. Neutron flux parameter of MNSRs are known to be stable, thus permitting the use of semi absolute Neutron Activation Analysis (NAA) [6-8].

NAA in its instrumental form (INAA), more especially with research reactors has some unique features which makes it attractive to use for routing analysis.

Some of the unique advantages that NAA has over the usual wet chemistry methods or the many spectrophotometric methods are:

 \succ Its sensitivity which depends on factors such as Neutron flux, sensitivity of detectors, time irradiated, neutron cross-section, etc.

 \succ It is a non-destructive process of determining chemical composition of the sample - since the sample is not changed in any manner by chemical manipulation or introduction.

 \succ It measures the total amount of the element in a material without regard to chemical or physical form, i.e. the samples do not have to be put in solution or vapourised. This is so because neutrons have no charge and can pass through most materials without difficulties.

 \succ The neutrons activate the nucleus of an atom and do not interact with the electron shell, and so detect the total elemental content regardless of oxidation state, chemical form or physical location.

 \succ It is highly precise and accurate by detection and measurement of characteristic emitted gamma radiation

Small amount of the sample (50mg) is sufficient for the analysis

> The technique is time efficient for a large number of samples can be irradiated at a given time and/or counted later on a given decay schedule.

Some other advantages are its multi-element capability, rapidity, reproducibility of result .

However, for many workers in the field a nuclear reactor is too expensive, instead it is common to use a neutron source which uses a combination of an alpha emitter and beryllium. These sources tend to be much weaker than reactors. Also, although the samples may become slightly radioactive in NAA, the radiation in the sample decreases with time until it reaches a state similar to which it was before the NAA is performed.

The MNSR in summary is a tank-pool type reactor which has a core with highly enriched uranium as fuel, light water as the moderator, and metallic beryllium as the reflector, it uses natural circulation for heat removal, negative reactivity temperature effect and very small excess reactivity. Its start-up and shut down is controlled by a micro computer; there are ten (10) irradiation sites in the MNSR. The temperature in each site is lower than 50°C even when in condition of full power of the reactor; the MNSR is also compact in structure.

This paper, therefore, examines the analysis and characterization of buffing wastes and discusses their effects on the Nigerian environment and suggests strategies for effective pollution control in the Nigerian leather industry.

MATERIALS AND METHODS

Buffing dust samples were collected from the tannery of the Federal College of Chemical and Leather Technology, Zaria, Nigeria. The buffing dust samples to be analyzed were dried then ground into a powdered form of 125µm grain size using an agate mortal and piston.

NIRR-1 which is a low-power nuclear reactor with highly enriched uranium as fuel, light water as moderator and beryllium as reflector was used for the analysis. The reactor's associated facility for radioactivity measurements are a gamma ray data acquisition system. It consists of a horizontal dip-stick High Purity Germanium (HPGs) detector with a relative efficiency of 10% at 1332.5 keV gamma ray line, MAESTRO emulation software compatible with the ADCAM[®] Multi-channel analyzer (MCA) card, associated electronic modules all made by EG & G ORTEC and a personal computer. The efficiency curves of the detector system at near and far source detector geometries have been determined by standard gamma-ray sources in the energy range of 59.5-2254 keV and extended to 4000KeV. For data processing gamma ray spectrum analysis software WINSPAN 2004 [9] developed at CIAE, Beijing, China was used. On the basis of the well known activation equation, the software requires that calibration factors be pre-determined by a multi-element standard reference material for elements of interest using adopted irradiation and counting regimes. The WINSPAN 2004 in addition to NAA calculations, perform peak analysis, remote control of MCA and other auxiliary functions such as efficiency calibration and nuclear data generation.

In this work, the certified reference material IAEA-Soil 7 was used to determine the calibration factors for all the element. For the determination of calibration factors sample aliquots of the standard of approximately 150mg was weighed and wrapped in polyethylene films. The polyethylene films and the rabbit capsules were cleaned by soaking in 1.1 HNO_3 for 3 days and washed with de-ionized water. Blank concentrations of all the elements were investigated using adopted procedures and found to be below the limits of detection of the set up for the polyethylene films.

For irradiation, two schemes (long and short) were adopted based on the half-life of the product radio nuclide. For elements leading to short-lived activation products the samples are each packed and sealed in 7cm^3 rabbit capsules and sent for irradiation in turn in an outer irradiation channel B₄ where the neutron spectrum is "soft" the choice of the outer irradiation channel is to eliminate corrections, notable Mg in the presence of P. This is due to the proximity of the inner channels of MNSRs to the core leading to relative higher ratio of fast-to-thermal neutron.

For elements leading to long-lived activation products, samples wrapped in polyethylene films were then irradiated for 6h in any of the small inner irradiation channels (i.e. A_1 , B_1 , B_2 and B_3) to take advantage of the maximum value of thermal neutron flux in the inner channels. The neutron flux variability over irradiation volume was determined experimentally to be less than 2% through the measurement of specific activities of irradiated Cu wires arranged axially and radially inside the vial. The stability of the neutron flux throughout the period of irradiation for the long irradiation was checked by monitoring the neutron flux reading of fission channel connected to the micro computer control system. The results indicate a stable neutron flux over irradiation period.

Radioactivity measurements of induced radio nuclides were performed by the PC-based gamma ray spectrometry set up. Following the short irradiation regime the first round of counting was performed for 10 min (i.e. S1) after a waiting time of 2-15 min. Samples were placed on a Plexiglass samples holder designated "H2" which corresponds to source-detector geometry of 5 cm. The second round of counting was also carried out for 10 min. following the short irradiation regime (i.e. S2) after a waiting period of 3-4 hr. Samples were counted on a Plexi-glass holder designated as "H1" corresponding to a source-detector geometry of 1 cm. The neutron flux setting for the short irradiation of the biological materials was raised to 5 x 10^{11} n/cm/s sensitivities for analysis of elements using procedures S1 and S2.

In the case of the long irradiation scheme, the first round of counting was carried out for 30 min following the long irradiation (i.e. L1) using the holder "H 1" after a waiting period counting was performed for 60 min (i.e. L2) after cooling time of 10-15 days. The samples were counted using the Plexi-glass holder "H1". The Choice of cooling time and sample detector geometry was such that detectors' dead time is controlled to be less than 10%.

A description of the irradiation and counting regime adopted for NIRR-1 facilities as well as radio nuclide of interest are given in Table 1.

Identification of gamma-ray products of radio nuclides through their energies and quantitative analysis of their concentrations were achieved using the gamma-ray spectrum analysis software WINSPAN 2004. For the biological material the certified reference material IAEA-359 (Cabbage) and IAEA-336 (Lichen) were chosen.

RESULTS AND DISCUSSION

The irradiation and counting schemes together with the WINSPAN multi-element gamma-ray analysis software have been validated using the standard reference materials.

Results printed in Table 1 give the routine irradiation and measuring regimes developed for NIRR-1 facilities. Table 2 gives comparison of certified values with our results in ppm or as indicated in %for certified biological reference material, IAEA-336 (Lichen) and IAEA-359 (Cabbage). Table 3 gives nuclear data and limits of detection for the elements of interest using adopted experimental conditions. Table 4 gives the analytical results of INNA of buffing dusts.

Table 1	Routine	irradiation	and mea	asuring r	egimes	developed	for	NIRR-1	facilities
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Neutronflux/irradiation channel	Procedure	T _{irr}	T_d	T _c	Activation products
1x10 ¹¹ n /cm ² s /outer irradiation	S1	2min	2-15min	10min	²⁸ A1, ²⁷ Mg, ³⁸ C1, ⁴⁹ Ca, ⁶⁶ Cu, ⁵¹ Ti, ⁵² V, ^{116m} In
channels (B4, A2)	S2	2min	3-4h	10min	²⁴ Na, ⁴² K, ¹⁶⁵ Dy, ⁵⁶ Mn, ^{152m} Eu
	L1	6h	4-5d	30min	239 Np (U), 72 Ga, 122 Sb
5x10 ¹¹ n /cm ² s /inner irradiation channels (B1,B2,B3, L2 and A1)	L2	6h	10-15d	60min	⁴⁶ Sc, ¹⁴¹ Ce, ⁶⁰ Co, ⁵¹ Cr, ¹³⁴ Cs, ¹⁵² Eu, ¹⁷⁷ Lu, ¹³¹ Ba, ⁸⁶ Rb, ¹⁸² Tb, ¹⁷⁵ Yb, ²³³ Pa (Th), ⁶⁵ Zn, ⁵⁹ Fe ^{, 181} Hf

It has been reported [10] that buffing dust generated by the leather industry is a proteinous solid waste impregnated with chromium, synthetic fat, oil, tanning agents and dye chemicals. In the present study, the characterization of buffing dust obtained from CHELTECH, Zaria using NIRR-1 has further revealed the elements present in buffing dust and their concentrations.

The results clearly indicate that chromium is significantly present (see table of result) in the buffing dust. This is obvious because majority of leathers are manufactured using chromium salt as the tanning agent. And according to Chronska and Przepiorkavska [11], buffing dust from chrome tanned leather is one of the difficult tannery wastes to manage. It is also hazardous to both human health and the environment. If Cr⁺⁶ is inhaled it causes cancer [12] and the issue in German regulations for example requires that any suspected carcinogen should not be present at higher than detectable levels. The result of this analysis suggests that chromium in leather and its manufacture is hazardous both to the operators and the environment. In the USA it was reported [13] that the Environmental Protection Agency had raised a serious concern over the dumping of leather waste (buffing dust, chrome shavings, etc) to an unsecured landfill. The fear is that Cr (III) might leak from a public landfill, enter a drinking water supply and be oxidized to Cr (VI) when the water is chlorinated. Therefore, the indiscriminate disposal of buffing dust from the leather industry in Nigeria and in the developing countries should be a thing of serious concern.

Element	IAEA-336(Lichen)	CV	IAEA-339 (Cabbage)	(CV)
	This work (TW)	CV	This Work (TW)	
Al	709±35	570-790	175±13	0.096-0.104
As	BDL	0.55-0.71	BDL	10.5-11.5
Ba	11.0 ± 1.3	5.3-7.5	BDL	-
Br	BDL	-	8.0±3.0	1.8-1.9
Ca (%)	BDL	1.11-1.45	1.97 ± 0.12	-
Ce	BDL	0.24-0.34	BDL	-
Со	BDL	0.89-1.23	BDL	1.24-1.36
Cr	BDL	0.097-0.123	BDL	-
Cs	BDL	144	BDL	78.6
Cu	BDL	0.019-0.027	BDL	-
Eu	BDL	0.038-0.048	BDL	0.014-0.15
Fe (%)	BDL	0.16-0.21	BDL	-
K (%)	0.66 ± 0.04	0.56-0.76	3.3 ± 0.4	-0.21-0.22
La	BDL	0.004-0.009	0.27 ± 0.05	31.3-32.5
Lu	BDL	-	BDL	567-601
La	61 <u>+</u> 2	56-70	BDL	-
Mg (%)	317 <u>+</u> 16	280-360	29.4 ± 0.8	-
Mn	BDL	1.54-1.98	676 ± 30	-
Na	BDL	0.063-0.083	BDL	-
Rb	0.16 <u>+</u> 0.03	0.15-0.17	BDL	-
SB	BDL	0.092-0.12	BDL	-
Sc	BDL	0.012-0.016	BDL	-
Sm	BDL	0.12-0.16	BDL	-
Tb	BDL	1.25-1.69	BDL	-
Th	BDL	0.025-0.049	BDL	-
V	BDL		BDL	-
Yb	BDL		BDL	-
Zn	BDL	27.0-33.8	BDL	-

Table 2. Comparison of certified values (CV) with our results in ppm or as indicated in % for the certified reference materials, IAEA-336 (Lichen) and IAEA 359 (Cabbage)

BDL-below detection limit.

The presence of Co, Cu, Cr and Zn in the sample analyzed could be attributed to the use of metal complex dyes [14] in leather manufacture. Generally, color fastness of leather is important especially when dealing with certain type of leathers such as washable/ clothing and upholstery leathers which should be resistant to dry cleaning solvents. Turner [15] outlined some leather metal complex dyestuffs with the percentages of the different elements as follows; CI Acid Black 76=1.7% Cu, CI Acid Yellow 243=1.3% Co and CI Acid Black 194=4.0% Cr.

The problem with the metals mentioned above is that whilst the dyestuffs themselves are not especially toxic/ harmful, they will add to the heavy metal load when the buffing dust is incinerated or spread on land. The reaction products of some of these elements present in the buffing dust could be most notably toxic. For example, Zn (with a concentration of 712 ppm) can form zinc oxide and on inhalation, the individual can develop Zn metal fume fever. This is an uncomfortable condition characterized by elevated temperature and "chills".

Arsenic with a concentration of 3.0 ppm (see table of results) in the buffing dust analyzed, can form a number of toxic compounds. The possibility of it forming the toxic +3 state oxide (As_2O_3) when absorbed through the lungs and intestines cannot be ruled out.

Target isotope	Product isotope by	Half-life	Gamma-energy	LOD (ppm)
²³ Na	²⁴ Na	14 96h	1368.60	40 (L1)
²⁶ Mg	27Mg	9.46min	1014.4	7250 (\$1)
²⁷ Δ1	²⁸ Δ1	2.24min	1778.99	17(S1)
³⁷ Cl	³⁸ Cl	37.24	1624.7	2900(S1)
41K	⁴² K	12 36h	1524.58	2400(S2)
45Sc	46Sc	83.81d	889.28	0.2(L2)
⁴⁸ Ca	⁴⁹ Ca	8.72min	3084.54	6600 (S1)
⁵⁰ Ti	⁵¹ Ti	5.76min	329.08	2500 (S1)
⁵⁰ Cr	⁵¹ Cr	27.7d	320.98	2300(31)
51V	⁵² V	3.75min	1434.08	15 (S1)
⁵⁵ Mn	⁵⁶ Mn	2.58h	846.76	0.9 (S2)
⁵⁸ Fe	⁵⁹ Fe	44.5d	1099.25	829 (L2)
⁵⁹ Co	⁶⁰ Co	5.27v	1173.2	3.0 (L2)
⁶⁵ Cu	⁶⁶ Cu	5.10min	1039.2	172 (S1)
⁶⁴ Zn	⁶⁵ Zn	243.9d	1115.55	120 (L2)
⁷¹ Ga	⁷² Ga	14.1h	834.1	1.0 (L1)
⁷⁵ As	⁷⁶ As	26.32h	559.10	1.2 (L1)
⁸¹ Br	⁸² Br	35.3h	776.5	3.0 (L1)
⁸⁵ Rb	⁸⁶ Rb	18.8d	1076.6	3.0 (L2)
¹¹⁵ In	^{116m} In	54.15min	1097.3	0.5 (S1)
¹²¹ Sb	¹²² Sb	64.8h	564.24	0.5 (L1)
¹³³ Cs	¹³⁴ Cs	2.06y	795.85	1.7 (L2)
¹³⁰ Ba	¹³¹ Ba	11.8d	496.3	264 (L2)
¹³⁹ La	¹⁴⁰ La	40.3h	1596.21	0.2 (L1)
¹⁴⁰ Ce	¹⁴¹ Ce	32.5d	145.44	14 (L2)
¹⁵¹ Eu	¹⁵² Eu	13.3y	1408.5	0.6 (L2)
¹⁵² Sm	¹⁵³ Sm	46.27h	103.18	0.1 (L1)
¹⁵⁹ Tb	¹⁶⁰ Tb	72.3d	879.38	1.1 (L2)
¹⁶⁴ Dy	¹⁶⁵ Dy	2.33h	94.70	0.7 (S2)
¹⁷⁴ Yb	¹⁷⁵ Yb	4.19d	396.33	0.9 (L1)
¹⁷⁶ Lu	¹⁷⁷ Lu	6.71d	208.36	0.1 (L2)
$^{180}{ m Hf}$	181 Hf	42.4d	482.2	1.1 (L2)
¹⁸¹ Ta	¹⁸² Ta	115d	1221.4	1.0 (L2)
¹⁹⁷ Au	¹⁹⁸ Au	2.7d	411.8	0.02 (L1)
²³² Th	²³³ Pa	27.00d	312.01	1.2 (L2)
²³⁸ U	²³⁹ Np	2.36d	277.60	1.5(L1)

Table 3. Nuclear data and limits of detection for the elements of interest using adopted experimental conditions

S1, *S2*, *L1* and *L2* represent Irradiation and counting schemes adopted for the respective element.

Bromine is another important element that was detected in the sample. Bromine is said to be a volatile dark red liquid that is toxic when inhaled or ingested. Like chlorine and fluoride, it is strongly irritating to the mucous tissue of the respiratory track and eyes and it can cause pulmonary edema [16]

Elements	Product isotope by (n, γ) reaction	Half-life	Gamma-energy (KeV)	Concentrations
Al	²⁸ A1	2.24min	1778.99	1.76±0.24%
As	⁷⁶ As	26.32h	559.10	3.5±0.2 ppm
Ba	¹³¹ Ba	11.8d	496.3	64±4 ppm
Br	⁸² Br	35.3h	776.5	3.1±02 ppm
Ca	⁴⁹ Ca	8.72min	3084.54	1.0±0.1ppm
Co	⁶⁰ Co	5.27y	1173.2	3.4±0.2 ppm
Cr	⁵¹ Cr	27.7d	320.98	0.5±0.02 %
Cs	¹³⁴ Cs	2.06y	795.85	0.23±0.05 ppm
Dy	¹⁶⁵ Dy	2.33h	94.70	BDL
Eu	¹⁵² Eu	13.3y	1408.5	0.06±0.01ppm
Fe	⁵⁹ Fe	44.5d	1099.25	4.3±0.1%
Hf	¹⁸¹ Hf	42.4d	482.2	BDL
K	42 K	12.36h	1524.58	0.55±0.08%
La	¹⁴⁰ La	40.3h	1596.21	11.5±0.3 ppm
Lu	¹⁷⁷ Lu	6.71d	208.36	0.03±0.005 ppm
Mg	²⁷ Mg	9.46min	1014.4	BDL
Mn	⁵⁶ Mn	2.58h	846.76	153±21 ppm
Na	²⁴ Na	14.96h	1368.60	1.27±0.03%
Rb	⁸⁶ Rb	18.8d	1076.6	24±2 ppm
Sb	¹²² Sb	64.8h	564.24	0.8±0.05 ppm
Sc	⁴⁶ Sc	83.81d	889.28	2.02±0.06 ppm
Sm	¹⁵³ Sm	46.27h	103.18	2.1±0.1 ppm
Та	¹⁸² Ta	115d	1221.4	BDL
Tb	¹⁶⁰ Tb	72.3d	879.38	NA
Th	²³³ Pa	27.00d	312.01	BDL
Ti	⁵¹ Ti	5.76min	329.08	BDL
U	²³⁹ Np	2.36d	277.60	BDL
V	⁵² V	3.75min	1434.08	BDL
Yb	¹⁷⁵ Yb	4.19d	396.33	BDL
Zn	⁶⁵ Zn	243.9d	1115.55	769±21 ppm

Table 4. Determined Elements and Concentrations of Buffing Dust using NIRR-1 Determined Elements and Concentrations of Buffing Dust using NIRR-1

BDL: - Below Detection Limit; NA: - Not Analysed.

CONCLUSION

The outcome of this work has indicated that buffing dust from tannery is characterized by wide range of elements that might likely pose health hazard to the tannery workers and impact negatively to the environment. Hence, to be able to reduce the environmental impact of buffing dust and other tannery wastes, the Leather Industry must design excellent sustainability/ environmental initiatives that would allow it to control and monitor the supply of all the materials and chemicals use for the manufacturing of their products in order to meet the international/ national regulations on control and prevention of pollution. Buffing dust should also be properly managed before disposal to the environment.

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