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ORIGINAL PAPER

Radiation activities and application of ionizing radiation on cultural heritage at ENEA Calliope gamma facility (Casaccia R. C., Rome, Italy)

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Abstract. Since the 1980s, research and qualification activities are being carried out at the ^{60}Co gamma Calliope plant, a pool-type irradiation facility located at the Research Centre ENEA-Casaccia (Rome, Italy). The Calliope facility is deeply involved in radiation processing research and on the evaluation and characterization of the effects induced by gamma radiation on materials for different applications (crystals, glasses, optical fibres, polymers and biological systems) and on devices to be used in hostile radiation environment such as nuclear plants, aerospace and high energy physics experiments. All the activities are carried out in the framework of international projects and collaboration with industries and research institutions. In the present work, particular attention will be paid to the cultural heritage activities performed at the Calliope facility, focused on two different aspects: (a) conservation and preservation by bio-deteriogen eradication in archived materials, and (b) consolidation and protection by degraded wooden and stone porous artefacts consolidation.

Keywords: cultural heritage • gamma radiation • irradiation plant • radiation effects • scintillators

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Introduction

Since the 1980s, the Calliope gamma irradiation facility (ENEA-Casaccia R. C., Rome, Italy) has been deeply involved in the qualification and research activities such as agricultural, radiation processing on industrial materials (polymers and optical fibres) and on devices to be used in hostile radiation environment (nuclear plants, aerospace experiments and high energy physics experiments), scintillating materials (crystals and glasses) as detectors in high energy and medium energy physics experiment, ionizing damage evaluation tests on electronic components, in the framework of international projects and collaboration with industries and research institutions [1].

Particular attention is paid to the effects of the irradiation parameters (absorbed dose, dose rate, temperature, atmosphere) on the irradiated material features, investigated by many different spectroscopic and luminescence techniques. Researches for conservation and preservation of cultural heritage archived materials (books, images) are related to the bio-deteriogen eradication assisted by gamma radiation and for assessment of recovery procedures, while consolidation and protection of wooden and stone porous artefacts are performed by radiation induced *in situ* polymerization of consolidant precursors.

Research and qualification activities at Calliope facility

The Calliope gamma irradiation facility, located at the Research Centre ENEA-Casaccia (Rome), is deeply involved in qualification and research activities, in the framework of international projects and collaborations with industries and research institutions [2].

Many research activities are focused on the investigation of gamma irradiation effects on chemical and physical properties of different materials, such as radiation detectors, scintillating crystals and glasses, for several applications (nuclear plants, aerospace, high energy physics experiments) [3–6]. Several investigations on $PbWO_4$ (PWO) scintillating crystals were performed at the Calliope facility in the framework of CMS-ECAL experiment at LHC (CERN, Geneva) [7] and a considerable improvement of the crystal radiation resistance was achieved by the optimization of the lead tungstate crystal doping with large and stable trivalent ions, such as La^{3+}, Lu^{3+}, Gd^{3+} and Y^{3+} [3, 8–12]. R&D studies were performed on doped and undoped cesium iodide crystals radiation hardness for the Belle II experiment at SuperKEKB (Japan), in order to up-grade the calorimeter to cope with the higher luminosity, pile-up and occupancy [13]. Optical coupling materials (optical grease, silicon and epoxy resins), usually applied to ensure good optical matching in the APD-scintillating crystal detection system, were investigated to establish their radiation hardness and stability under gamma and neutron irradiation [13].

Researches were focused on the optical components operating in space during interplanetary missions, since the exposition to fluxes of energetic particles may deteriorate their performance. In order to simulate the hostile radiation environment, gamma irradiation tests were carried out and the induced optical damage in the UV-VIS-NIR spectral region and the recovery after the end of irradiation were investigated [14, 15].

Synthetic and natural polymeric materials used in many field (e.g., nuclear and space application, medical devices, food packaging, cultural heritage, etc.) are studied in terms of gamma irradiation induced processes (cross-linking and degradation). Their behaviour is evaluated in different atmospheric conditions (air, vacuum or inert gases), paying particular attention to the irreversible modification occurring during or after the end of gamma irradiation [6, 16–19]. Among the ongoing research activities at the Calliope facility, investigations are related to the rad-hard packaging for electronic components and devices to be used in radiation-rich environment. The activities are focused on the qualification of standard packaging materials (metals, metal-glass systems, glass-ceramic systems, ceramic multilayer, etc.) on the study of new materials (polymeric compounds and composites, metal nitrides) and technologies.

In recent years, the application of biopolymers in food industry and packaging has significantly increased. Ionizing radiation can be used to improve the properties of biopolymer-based films, mainly composed by polysaccharides and proteins [20]. Ongoing researches, carried out by spectroscopic and luminescence analyses, are focused on the investigation of gamma induced effects on these biopolymers. Agriculture and the environmental biological activities, such as biological control of pests assisted by gamma irradiation (SIT, Sterile Insect Technique) and agricultural product treatments, are also carried out [1].

Qualification tests are performed, in compliance with the international standard specifications, on electronic components and devices for application in hostile environments such as nuclear plant and aerospace, and on concrete matrices for nuclear waste disposal and storage [21]. The qualifications of electronic devices are tested according to MIL-STD-883 and/or ESA/SCC BASIC Specifications No. 22900 procedures [22, 23]. Recently, several qualification activities for the International Thermonuclear Experimental Reactor Project (ITER) were carried out at very high absorbed doses (up to 4–5 MGy) on components and devices, such as piezo-motors and optical components for the in-vessel viewing system (IVVS), scintillators for the radial neutron camera (synthetic diamonds, crystals and plastics) [24].

Calliope irradiation facility

The Calliope facility is a pool-type irradiation facility equipped with a ^{60}Co γ source in a high volume ($7 \times 6 \times 3.9$ m^3) shielded cell (Fig. 1). The emitted radiation consists of two γ photons of 1.173 and 1.332 MeV emitted in coincidence, with the mean photon energy of 1.25 MeV. The Calliope maximum licensed activity is 3.7×10^{15} Bq (100 kCi). A dedicated scheduling and reporting software system has been set up at the Calliope facility [1].

The Calliope facility is equipped with dosimetric laboratory. Fricke absolute dosimetry (in the range of 20–400 Gy), Red Perspex (in the range of 5–40 kGy), thermoluminescent dosimetry (very low dose, <20 Gy) and EPR-alanine dosimeter (about 500 kGy) are used as dosimetric methods [1, 25].

The characterization of the irradiation effects is performed at the Calliope laboratories, equipped with several instruments for the evaluation of optical (UV-VIS and FTIR spectrophotometer, luminescence measurements) and spectroscopic (ESR spectrometer, working in the temperature range 77–298 K and in a different atmosphere such as O_2, CO, N_2, Ar) behaviour of materials under irradiation [1]. Viscosity measurements for the determination of polymerization degree are carried out.

Application of ionizing radiation for cultural heritage

In the context of the peaceful use of nuclear and radiation technologies, nuclear techniques, with special emphasis on gamma radiation treatment, are suggested for the characterization and preservation of cultural heritage artefacts including

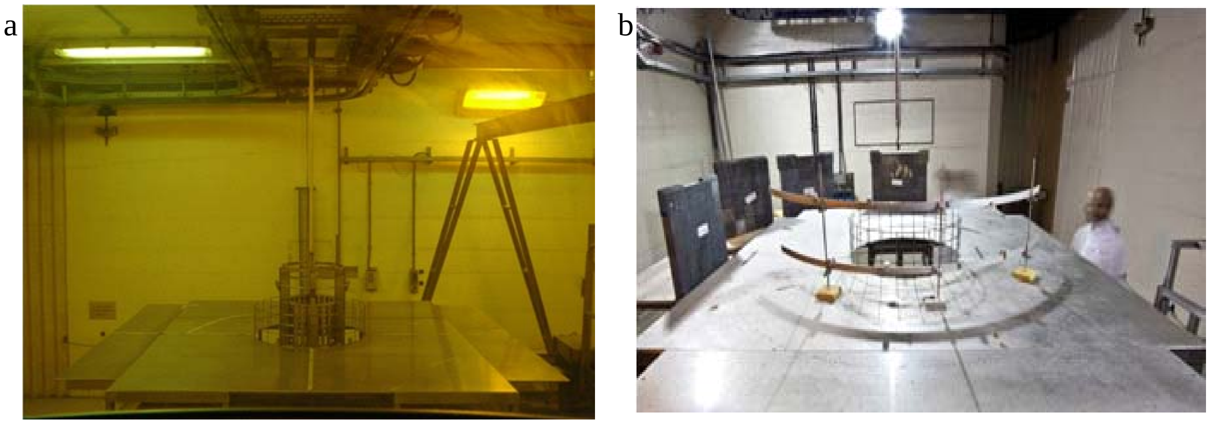

Fig. 1. (a) Steel platform covering the pool, as seen through the yellow lead glass window of the control room. (b) Details of movable support for dosimetric measurements.

eradication of insects, disinfection of microorganisms and consolidation of degraded materials with radiation-curing resins [26–28]. In the last decades, it was demonstrated that nuclear techniques are exceptionally suitable not only for non-destructive characterization of cultural heritage artefacts, but also in support of their conservation and restoration, as well as for their preservation through the use of radiation treatment.

The physical properties of electromagnetic radiation (i.e., gamma rays from ^{60}Co source) allow them to penetrate materials with extremely harmful effect on living organisms. By the irreversible denaturation or cleavage of nucleic acids, organisms and microorganisms present on the surface and in the bulk of the irradiated objects, are simultaneously and indiscriminately devitalized [29]. The attack of microorganisms and/or insects, the so-called bio-deteriogens agents, can indeed cause a rapid, disastrous and often irreversible damages to cultural materials containing cellulose, starchy material, albumen, glue, vegetable proteins, dyestuffs, pigments, dispersing agents, and other additives that provide food sources for the infesting organisms. The importance of bio-deterioration is underlined by the high frequency of the most harmful natural calamities' occurrence (i.e., floods, earthquakes, etc.) that can affect books and archives, inducing infection and damaging a great amount of them. Moreover, bio-deteriogens can cause severe health problems for restorers, archivists or librarians.

Consolidation and surface protection of degraded wooden or stone artefacts (i.e., porous materials) represent one of the most interesting challenges in the cultural heritage [30]. As it is known, many different atmospheric and biological agents, such as humidity, temperature leaps, biological growth or chemical attack, induce severe and somewhat irreversible degradation phenomena on these kind of materials.

Regarding stone artefacts and architectural structures, the main degradation effect consists of a sensible loss of their mechanical properties, increasing their fragility by mortar pulverization or causing great modification of surface aesthetical features. On the other hand, ancient wooden cultural objects undergo many different chemical and physical changes, due to the biodegradable nature of the material itself. In this perspective, the use of high-penetrating ionizing radiation such as gamma rays is extremely suitable to induce *in situ* polymerization of synthetic consolidating agents.

By gamma radiation, it is possible to: (i) remove bio-deteriorating agents; (ii) stop the ongoing destructive process; (iii) restore the object of cultural value. Other important advantages that can be mentioned are: physical method due to which no toxic or radioactive residues remain in treated item or environment; due to the high penetration power of gamma radiation, large amounts of bio-deteriorated objects can be quickly treated (probably the only method in case of emergency).

The evaluation of the often irreversible physicochemical modifications induced by ionizing radiation on treated materials, namely side-effects, represents an important goal to guarantee the safeguard of the treated artefacts. One of the main obstacle to the diffusion of nuclear technology is effectively the negative effect that ionizing radiation such as gamma rays can induce in the polymeric materials (i.e., paper, wood, resins) [31–33]. This effect consists of the de-polymerization of the polymeric network, proportional to the absorbed radiation. It occurs either directly on the macromolecule intermolecular bonds, or indirectly through a chemical effect mediated by free radicals that could cause post-irradiation changes. The direct effect simultaneously causes a three-dimensional cross-linking and the breaking of the chains (i.e., bond β-glycosidic cleavage on cellulose, basic constituent of paper and wooden artefacts), measurable as a modification of the polymerization degree (DP), that exponentially decays with the increase of the absorbed radiation dose [31, 34, 35]. Although both these processes occur at the same time, cross-linking prevails at low absorbed doses while degradation (i.e., bonds rupture) becomes significant with the increase of the irradiation dose. Finally, while cross-linking improves the mechanical properties of materials, the chains breaking acts on the contrary, with dominant effect, over around 10 kGy [16].

Free radicals, very unstable and energetic short--life species (10^{-3} s), are also responsible for post--irradiation effects in the long-term. Throughout the indirect action of radiation induced free radicals, a key role is played by the oxygen present in the air during irradiation due to the oxidative degradation phenomenon.

Since this process is related to the oxygen diffusion rate inside the material, it is clear that considering the same absorbed dose value, the longer the exposure time (low dose rate), the greater are the resulting damages [18]. However, since the production of free radicals is proportional to the radiation dose, it is possible to minimize the oxygen degradation operating at dose rate as high as possible (but compatible with the treated material) or performing the irradiation tests in inert atmosphere or in vacuum [36].

Considering the doses suggested for treatment of disinfestation, it was demonstrated that the radical degradation effects in these conditions are not significant [26].

Side-effect evaluation can be carried out by identifying one or several physical, chemical or structural parameters that can be directly measured after appropriate definition of methodical procedures. For this purpose, many destructive or non-destructive techniques are employed to characterize the radiation damage on the treated materials [28].

Researches on conservation/preservation and on consolidation/protection

In ENEA laboratories, gamma radiation has been used for different applications, often associated to accelerated ageing or to modified atmosphere, to verify the irradiation side-effects over time on several materials (pure cellulose, permanent paper, inks, etc. [37, 38]. Moreover, further experimental trials were carried out to investigate whether, and up to which level, the chemical and physical changes induced in paper (newspaper, magazine, permanent paper and cellulose) by irradiation and/or ageing could negatively predispose this material, once treated, to the attack of destructive insects and microscopic fungi, increasing their harmfulness [39, 40]. The results of all testing activities lead to the assessment that the ionizing radiation treatment is extremely efficient for the disinfestations against harmful insects and for disinfection against microfungi. Moreover, using the necessary dose for an efficient immediate lethal treatment for insects (2–3 kGy) and microfungi (3–8 kGy), no significant harmful effect has occurred on the mechanical and physical properties of pure cellulose and of paper, on printing inks, on the vulnerability of treated material once subjected to infestation, as well as to infection due to bio-deteriorating agents [41]. In addition, the results have demonstrated that damage caused by organisms is strictly related to the state of degradation of cellulose and it is dependent on the absorbed dose range: the microbial population decreases, with an exponential or sigmoidal dependence, as the gamma rays dosage increase together with the polymerization degree of cellulose molecule [42, 43]. This phenomenon was negligible at the recommended treatment dosage (3–5 kGy); and in any case, it did not alter the essential mechanical functional characteristics of the paper.

With the same intent, studies have been extended to photographic material (developing out paper sensitized with gelatin silver print, ILFORD-ILFOBROM 2.1 P) and results put in evidence that no significant modifications, both of the growth of microfungi (*Penicillium chrysogenum*) and of the erosive action by chewing insects (*Blaptica dubia*), occur in the analysed photographic paper irradiated at 3 kGy, with respect to non-irradiated paper (Figs. 2 and 3) [28].

Moreover, other experimental trials have been carried out to verify if gamma rays could change the fastness of different colour dyes or pigment particles either suspended in the emulsion or added directly by the photographer. Results lead to the conclusion that the colour fastness of the treated photographic prints are not influenced by gamma radiation till 10 kGy [28].

With the aim to evaluate the dose rate and environmental atmosphere influence on Whatman paper, ongoing biological analysis are performed to investigate if the radio-induced effects can increase bio-deteriogens' harmfulness on treated papers. At the same time, irradiation side-effects and environmental influence on paper will be evaluated by chemical and physical analyses.

In paper structure, water plays a central role allowing the stabilization of the cellulose matrix

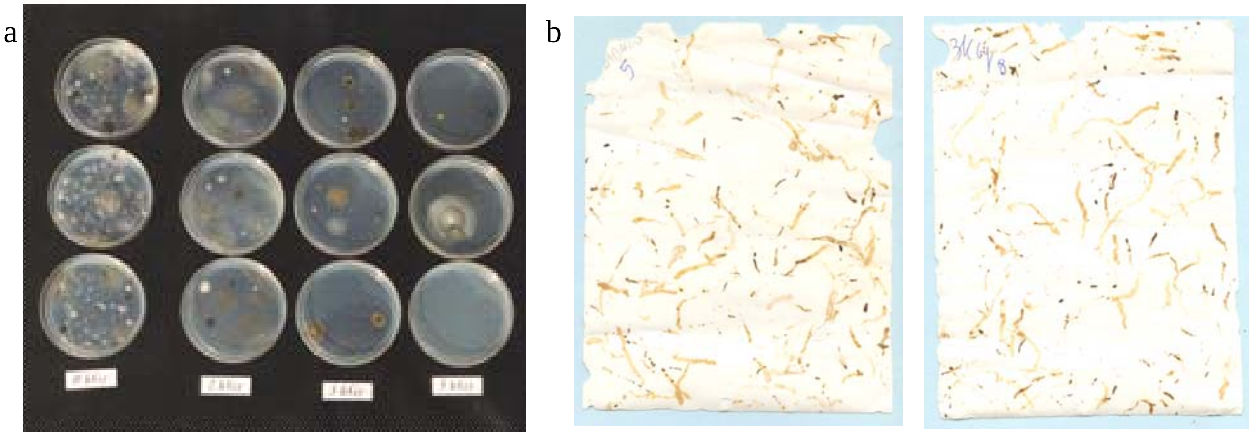

Fig. 2. (a) Microscopic moulds (mainly *Penicillium*): left to right: not irradiated and irradiated at 2, 3 and 5 kGy [28]; (b) damages by *Blaptica dubia* feeding on photographic paper samples: left, not irradiated; right, irradiated at 3 kGy [28].

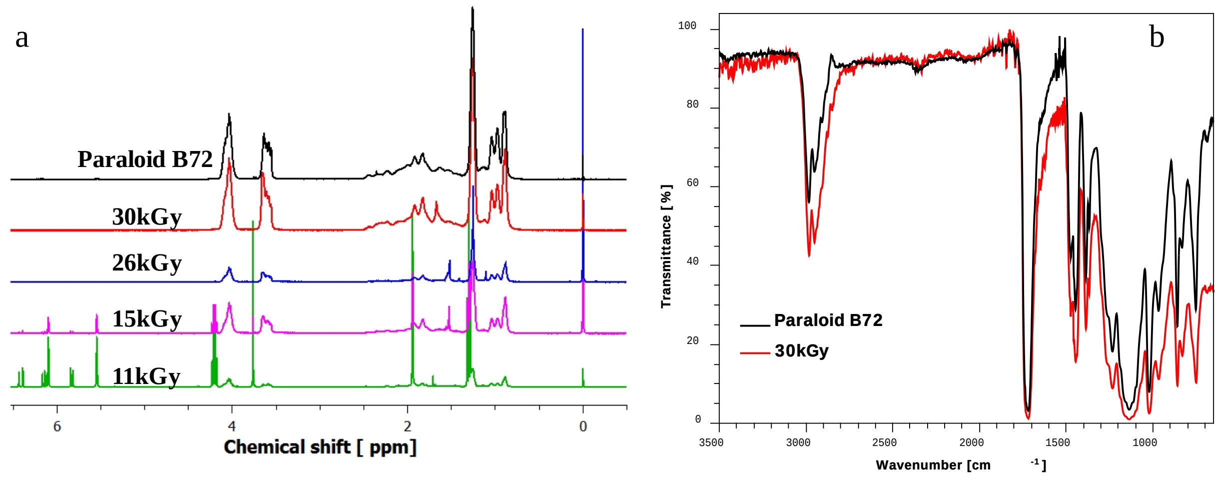

Fig. 3. (a) ^{1}H-NMR spectra of methyl acrylate- ethyl methacrylate co-polymers at different absorbed doses and of Paraloid B72; (b) FTIR-ATR spectra of co-polymer irradiated at absorbed dose of 30 kGy and of Paraloid B72 [39].

and determining most of its mechanical properties. Using gamma irradiation to induce paper ageing, pure cellulose Whatman paper was characterized to get information about siting and dynamics of water rearrangement when cross-linking and degradation of cellulose occur. By NMR 2D technique, a two-site (low- and high-mobility) residence model for water in the ultrastructure of cellulose was demonstrated. When cross-linking and degradation of cellulose occur, a significant fraction of water appears confined into low-mobility sites of the cellulose matrix [16].

Further investigation regarding the evaluation of gamma irradiation side effects on paper submitted to irradiation in the dose range 0–1000 kGy, paying particular attention to the low absorbed doses (up to 10 kGy), are of great interest for cultural heritage applications. The structural modifications of cellulose, that is, cross-linking and degradation processes, were studied by thermal analyses (TG-DTG) and Fourier-transform infrared spectroscopy (FTIR) and allowed a complete characterization of the cellulose-moisture interaction [17].

Ongoing activities are focused on the gamma irradiation *in situ* polymerization of methyl acrylate (MA) and ethyl methacrylate (EMA) monomeric solutions to obtain a co-polymer with similar characteristics to those of one of the most used consolidating products (Paraloid B72) in cultural heritage [44]. The optimization of the polymerization dose rate and absorbed dose was carried out, verifying, by means of ^{1}H-NMR and FTIR-ATR measurements, the production of same chemical structure of the sample irradiated at 30 kGy and of Paraloid B72 (Fig. 3), as reported in our research paper [45].

Conclusion

An overview of the research and qualification activities performed at the Calliope gamma irradiation facility (ENEA-Casaccia R.C., Rome, Italy) are described in this work. Specific attention is devoted to the application of ionizing radiation on the cultural heritage, for the conservation, preservation and for the consolidation and protection of the artefacts. Despite much work being done, it is still necessary to increase the knowledge on the advantages and limitations of nuclear technology for cultural heritage application. Besides, the statement of well-defined irradiation condition (in term of irradiation dose and dose rate, environmental atmosphere, pre-treatment of the cultural heritage object) and the proposal of shared guidelines are extremely desirable.

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