Oxygen absorption for the protection of archaeological iron: improving maintenance *

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The goal of this project was to the examine the efficiency of the Revolutionary Preservation System [RP System] oxygen absorbers manufactured by Mitsubishi Gas Chemical Company, Inc. for both the temporary storage of freshly excavated material and for the long-term storage of previously excavated materials. This paper reports on a new series of anoxic tests by the Conservation Department of the Japanese Institute of Anatolian Archaeology (JIAA) in Kaman, Turkey, using a MOCON OpTech-O_2 Platinum oxygen meter (+/-0.015% O_2 precision) to monitor the oxygen content in Escal test bags. Two test periods were monitored; four days for freshly excavated materials and 12 months for long-term storage performance. The oxygen levels targeted were <0.1% based on the color change of the RPS oxygen indicator, 0.015% based on the precision of the oxygen meter, and 0% as the ultimate target achieved by the RP System scavengers. The time required for the reduction of the oxygen content to 0% (+/-0.015% O_2) was monitored with the aim of placing freshly-excavated iron in anoxic storage as quickly as possible to prevent corrosion. Sixteen tests were run with RP-A and RP-K to assess oxygen absorption and with RP-A and silica gel to assess desiccation. During the duration of the tests the bags were kept in uncontrolled climatic conditions typical of archaeological storage facilities. The variables tested on oxygen absorption were the ratio of RP-A and RP-K scavenger weight to air volume, clipped versus heat-sealed Escal bags, and the impact of uncleaned archaeological iron. The RP System plastic clips were tested for temporary storage and were compared to the more permanent, heat-sealed method. RH was measured in a select number of test bags with dataloggers or humidity indicator cards. Complete results from the tests are presented regarding the use of the RP System for the storage of archaeological iron.

Keywords: relative humidity/oxygen/scavenger/Revolutionary Preservation System/Japanese Institute of Anatolian Archaeology (JIAA)/Turkey

Research Aim

The ultimate goal is the safe storage of archaeological iron by eliminating oxygen. Since the RP System oxygen scavengers are capable of achieving 0% O_2 (+/-0.015% O_2 oxygen meter precision), this is the ultimate target to be obtained as quickly as possible for the freshly excavated iron and to be maintained for as long as possible for all iron. Tests published in 2013 of the anoxic and desiccation properties of RP System RP-A scavengers and silica gel

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for the storage of archaeological iron resulted in the following recommendations: 1) determine a means more accurate than the RP System oxygen eye indicators for monitoring oxygen depletion, 2) carry out further studies to monitor oxygen depletion until the lowest oxygen level is reached by RP-A and RP-K and 3) test the impact of increasing the ratio of RP-A and RP-K scavenger weight to air volume (Paterakis and Mariano 2013). New tests of the RP System were carried out for both the temporary storage of freshly excavated material, before and after cleaning, and for the long-term storage of previously excavated materials. For this reason two test periods for were monitored; four days for freshly excavated materials and 12 months for long-term storage performance. The RP System plastic clips were tested for temporary anoxic storage and were compared to the more permanent, heat-sealed method. Sixteen tests were run with RP-A and RP-K to assess oxygen absorption and with RP-A and silica gel to assess desiccation to achieve these research aims. Test variables included the ratio of oxygen scavenger weight and silica gel weight to the volume of the test bags, the method of closing the Escal bags (clip versus heat-seal), and the influence of metal coupon corrosion monitors and uncleaned archaeological iron artifacts. The relative humidity was monitored with dataloggers in some tests and with humidity indicator strips in other tests. The oxygen level in the bags was monitored with a MOCON OpTech-O_2 Platinum oxygen meter (+/-0.015% O_2 precision).

Introduction

The Japanese Institute of Anatolian Archaeology (JIAA) collection at Kaman in Turkey holds thousands of iron objects that have been excavated since 1986. Since 2009 two new sites have been added to the JIAA's excavation roster increasing the rate of acquisition of iron artifacts. Encouraging results from initial tests of the anoxic and desiccation properties of RP-A scavengers in Escal bags for the storage of archaeological iron prompted further testing (Paterakis & Hickey-Friedman, 2011; Paterakis & Mariano 2013). The RP System may be used to prevent the corrosion of bronze and iron, two archaeological metals prone to chloride contamination and sensitivity to moisture. It has been suggested that RP-K scavengers may protect damp and structurally weakened iron from cracking upon drying by removing oxygen without altering the RH (Greiff & Bach 2000; Maekawa & Elert 2003). The efficiency of RP-K scavengers as oxygen absorbers without altering the RH was examined to assess their applicability for the storage of archaeological iron. Since the RP-K scavengers do not absorb moisture, oxygen reduction rate is evermore important for stabilizing iron.

Experimental

A new series of sixteen tests comprising five groups was run by the Conservation Department of the Japanese Institute of Anatolian Archaeology (JIAA) in Kaman, Turkey (Table 1). The MOCON OpTech-O_2 Platinum oxygen meter was used to monitor the oxygen content in Escal test bags. A temporary sensor was attached to the inside of each bag with the manufacturer's proprietary contact adhesive and the oxygen content was read directly through the Escal without piercing the bag (Figure 1). Readings were taken every hour (with the exception of hour two) for the first 10 hours of the tests. Two RPS oxygen indicator eyes were enclosed in each test bag for comparison with the oxygen meter readings. The RPS oxygen indicator eyes turn a pink color when the O_2 drops below 0.1% and a purple color when the O_2 exceeds 0.5% (Elert and Maekawa 2000; Guggenheimer 2006). RH was measured in a select number of test bags with dataloggers and in other tests with humidity indicator cards. Four tests were run for four days and 12 tests ran for 12 months. Each test was run in triplicate to produce averaged results from a total of 48 test bags. The bags were kept in uncontrolled climatic conditions throughout the test periods. The bag dimensions in the RP-A tests were 22 x 20 x 3 cm and the bag dimensions in the RP-K tests were 15.5 x 14 x 3 cm. The manufacturer's recommendations were followed to determine 3 RP-5A scavengers and 2 RP-3K scavengers as the suitable number based on bag size and volume. RP-A and RP-K scavengers were tested for their influence on the rate of oxygen absorption by varying the ratio of scavenger weight to volume of the test bag. RP-A scavengers and cobalt-indicating silica gel were tested for their influence on the rate of moisture and oxygen absorption by varying the ratio of scavenger and desiccant weight to volume of the test bag. Other variables introduced were clamped versus heat-sealed Escal bags and the impact of metal coupon corrosion monitors and uncleaned archaeological iron.

Table 1 Description of Tests and Rate of Oxygen Reduction

(averages are calculated for the first ten hours of testing, those tests not reaching the specified oxygen level of < 0.1%, 0.015%, or 0% are not averaged)

Group	Test	Oxygen Absorber and Desiccant	RH	Hours required to reach < 0.1% O_2[1]	Hours required to reach 0.015% O_2[2]	Hours required to reach 0% O_2	Time	Bag
ONE	1	RP-5A x 1	Rotronic HL-20 data-logger	7.7 24° C	1A, 1B, 1C=>10<22 23.5° C	1A=25, 1B, 1C= >10<22 23.5° C	12 months	Heat-sealed
	2	RP-5A x 3	Rotronic HL-20 data-logger	3.3 24° C	6.3 24° C	7.7 24° C	12 months	Heat-sealed

	3	RP-5A x 5	na	3 24° C	3A=7, 3B=8 3C=>10<22 24° C	3A=10 3B,3C=>10 <22 24° C	12 months	Heat-sealed
	5	Control	na	na	na	na	12 months 23°-25° C range 1st 24 hours	Heat-sealed
TWO	6	RP-5A x 3	RH card	3 24° C	6A, 6B=>10 < 22 6C=4 24° C	6A, 6B=>10 < 22 6C=6 24° C	4 days	Heat-sealed
	7	Cobalt indicating silica gel (100 g)	RH card	na	na	na	4 days 23°-25° C range 1st 24 hours	Heat-sealed
	8	RP-5A x 3 Cobalt indicating silica gel (100 g)	RH card	4 23.5° C	8A, 8B, 8C =>10 < 22 24° C	8A, 8B, 8C =>10 < 22 24° C	4 days	Heat-sealed
	9	RP-5A x 3 Cobalt indicating silica gel (100 g) Pb and Cu test coupons	RH card	6 24° C	9A=never 9B=>9<22 9C=22 23° C	9A=never 9B=22 9C=22 23° C	4 days	Heat-sealed
THREE	10	RP-3K x 1	HOBO U10 data-logger	8.6 24° C	10A=>10< 23 10B, 10C=9 24° C	10A=>10< 23 10B, 10C=9 24° C	12 months	Heat-sealed
	11	RP-3K x 2	HOBO U10 data-logger	6	8	11A, 11C => 10 < 23 11B=6	12 months	Heat-sealed

				24° C	24° C	24° C		
FOUR	12	RP-5A x 1 Uncleaned iron artifact	RH card	9 24° C	23 23.5° C	24 24° C	12 months	Heat-sealed
	13	RP-5A x 3 Uncleaned iron artifact	RH card	13A=7 13B=>9 <19 13C=9 23.5° C	13A=22 13B=54 13C=>9<19 23.5° C	13A=22 13B=54 13C=>9< 19 23.5° C	12 months	Heat-sealed
FIVE	4	Control	na	na	na	na	12 months 23°-25° C range 1st 72 hours	Clipped
	14	RP-5A x 1	na	14A=9 14B=>9 < 19 14C=6 23.5° C	14A=43 14B=22 14C=>19<22 23° C	14A=>43<56 14B=25 14C=22 23° C	12 months	Clipped
	15	RP-5A x 3	na	3.3 23.5° C	11.7 23.5° C	15A=>19<22 15B=22 15C=>28<43 23° C	12 months	Clipped
	16	RP-5A x 5	na	2 23.5° C	4.3 24° C	6 24° C	12 months	Clipped

[1] from 0% to < 0.1% O_2 the RPS oxygen indicator eye is a pink color

[2] +/- 0.015% O_2 is the precision of the MOCON OpTech®-O_2 Platinum oxygen meter
na = not applicable

Figure 1 reading oxygen content from the sensor in test bag with the MOCON OpTech-O_2 Platinum oxygen meter ©Middle East Culture Center in Japan

Figure 2 Escal test bag 16A sealed with plastic clip provided by the manufacturer ©Middle East Culture Center in Japan

Figure 3 closing Escal bag with FoodSaver V3240 Vacuum Heat-Sealer ©Middle East Culture Center in Japan

Description of Tests

The 4-day tests were subject to a surrounding minimum and maximum RH and temperature of 31% to 51% RH and 24° to 28° C. The RH and temperature in the storeroom during the 12-month tests ranged from 32% to 82% RH and - 2° C to 28° C.

Group One examined the influence of the ratio of oxygen scavenger weight to bag volume on the oxygen and moisture absorbing properties of RP-5A. Tests 1, 2, and 3 contained 1, 3, and 5 RP-5A scavengers respectively. RH was monitored in one Test bag 1A and in Test bag 2A with a Rotronic HygroLog HL-20 datalogger (± 0.8% - 1.3% RH, ± 0.2° C accuracy).

Group Two examined the influence of cobalt-indicating silica gel on the oxygen and moisture absorption of RP-5A in four tests. Three of the four tests (Tests 7, 8, 9) included 100 grams of cobalt-indicating silica gel. Tests 6, 8, and 9 contained 3 RP-5A scavengers (manufacturer's recommended amount). Test 9 also contained copper and lead corrosion test coupons. RH was monitored with a cobalt-containing humidity indicator card in each test.

Group Three tested the impact of the ratio of the RP-3K scavenger weight to bag volume on oxygen absorption. Test 11 held two RP-3K scavengers (manufacturer's recommended amount) and Test 10 held one RP-3K scavenger. RH was monitored in the Test bag 10A and Test bag 11A with a Hobo U10 datalogger (± 3.5 % RH, ± 0.53° C accuracy).

The Group Four tests included one freshly excavated and uncleaned iron object of similar dimension in each. Test 12 contained 1 RP-5A scavenger and Test 13 contained 3 RP-5A scavengers (manufacturer's recommended amount). The RH was not monitored in these tests.

Group Five compared closing the Escal bags with plastic clips supplied by the manufacturer versus heat-sealing (Figure 2). A FoodSaver V3240 Vacuum Sealer was used to heat-seal the bags by creating two parallel 3 mm wide seams on both cut ends of the Escal roll without utilizing the vacuum function (Figure 3). Tests 14, 15, and 16 contained 1, 3, and 5 RP-5A scavengers respectively. The RH was not monitored in these tests.

Results of Tests

The rate of oxygen depletion was measured hourly for the first 10 hours (except for hour two) of the tests (Figure 4). By 10 hours the oxygen had been reduced to very low levels and completely removed in some tests (Table 1). The greatest rate change in RH in Group One and Group Three also occurred during the first hours of the tests.

Group One tests demonstrated that increasing the ratio of RP-5A scavenger weight to bag volume results in increased rate of oxygen and moisture absorption. Exceeding the manufacturer's recommended number of scavengers based on bag volume was found to hinder the rate of absorption of oxygen from 0.1% to 0% O_2, which was unexpected (Table 1). The initial desiccating properties of one RP-5A (Test 1) and three RP-5A scavengers (Test 2) (the manufacturer's recommended amount) were equal reaching 35% RH (26° C) between the first and second hour of the tests (Figure 5). Subsequently, the one RP-5A scavenger (Test 1) reached 10% RH (24° C) after 64 hours, not keeping pace with three RP-5A scavengers (Test 2) that reached 10% RH (25° C) after 12 hours. 0% RH (22° C) was achieved by three scavengers (Test 2) in 403 hours and by one scavenger (Test 1) in 864 hours. It is interesting to note that in both tests the RH was 15% (24.5° C) when 0% O_2 was reached. While the temperature in the test bags mimicked the temperature changes in the surrounding storeroom, once the RH in the bags reached 0% the RP-A maintained the RH in the bag at a constant 0% RH for the duration of the 12-

month tests (despite fluctuations in the storeroom exceeding 20% RH in a 24 hour period).

In Group Two the cobalt-indicating silica gel and humidity indicator cards were found to inhibit slightly the absorption of O_2 down to <0.1% O_2 (24° C) in Tests 6 and 8 (Figure 6). The copper and lead test coupons in Test 9 slowed the rate of oxygen absorption to 0.1% <O_2 (24° C) (Figure 6). Although no visible changes to the coupons were evident, this reduced oxygen absorption rate may be attributed to slight corrosion of the test coupons as indicated by a weight gain of 0.31% (Cu) and 1.15% (Pb).

Group Three tests demonstrated that increasing the ratio of RP-3K scavenger weight to bag volume increased the rate of oxygen absorption (Figure 7). The RP-3K affected the RH in similar ways in both tests; the rate of RH change was the same in the first two hours of the tests culminating with 30% RH (26° C) in Test 11 and 32% RH (26° C) in Test 10 (Figure 8). This difference falls within the ±3.5% RH accuracy of the HOBO U10 dataloggers used to monitor these tests. Two RP-3K scavengers (manufacturer's recommended amount) in Test 11 maintained a slightly lower RH, 4% on average, throughout the duration of the tests which again may be attributed to the ± 3.5% RH accuracy of the HOBO U10 datalogger. Stabilization of the RH was achieved sooner (4 hours) than complete oxygen absorption (9 hours). The RH in both tests exhibited a maximum fluctuation of 10% in spite of extreme fluctuations in the storeroom. The RH coincided with general cooling and warming trends, with only small fluctuations occurring in the test bags while large fluctuations occurred in the storeroom. For example, in a 24-hour period from September 27 to 28, 2014, the RH in the storeroom fluctuated by ca. 24% (17 – 21 C°), while in Test 10A the RH remained constant at 33.5 % (18 – 20 C°) and in Test 11A at 30.5% (17 – 20 C°).

The Group Four tests showed that the uncleaned archaeological iron had a major impact on oxygen absorption by delaying the reduction of oxygen to < 0.1% O_2 from 3 hours in Test 2 (Group One) (24° C) to > 10 hours in Test 13 (Group Four) (23° C) (Figure 9). The variation in rate of oxygen reduction in Test 13 is attributed to diverse corrosion conditions and moisture contents of the uncleaned iron objects (Table 1). The average weight change of the iron objects was a loss in Test 12 of – 1.12% and in Test 13 of – 2.21%. The loss in weight may be attributed to the desiccation of the iron corrosion products and the superior weight loss in Test 13 may be attributed to the larger scavenger weight to volume ratio.

Group Five tests demonstrated that the rate of oxygen absorption was a bit slower and more erratic in the clipped bags than in the heat-sealed bags (Figure 10). However, the holding power of the clipped and heat-sealed bags over 12 months was excellent and maintained a constant 0% RH and 0% O_2 once these levels were reached.

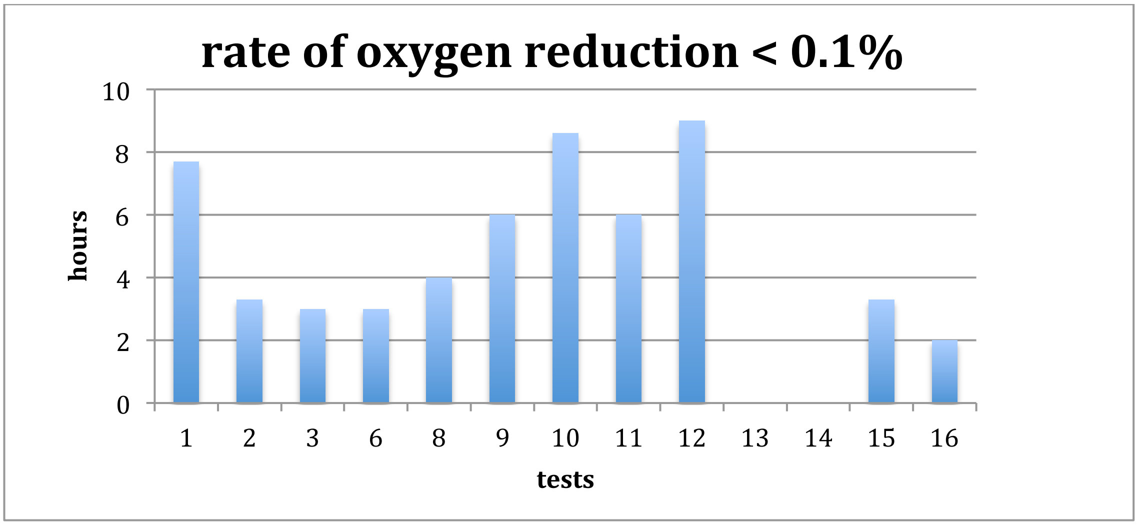

Figure 4 rate of oxygen reduction to less than 0.1% O_2

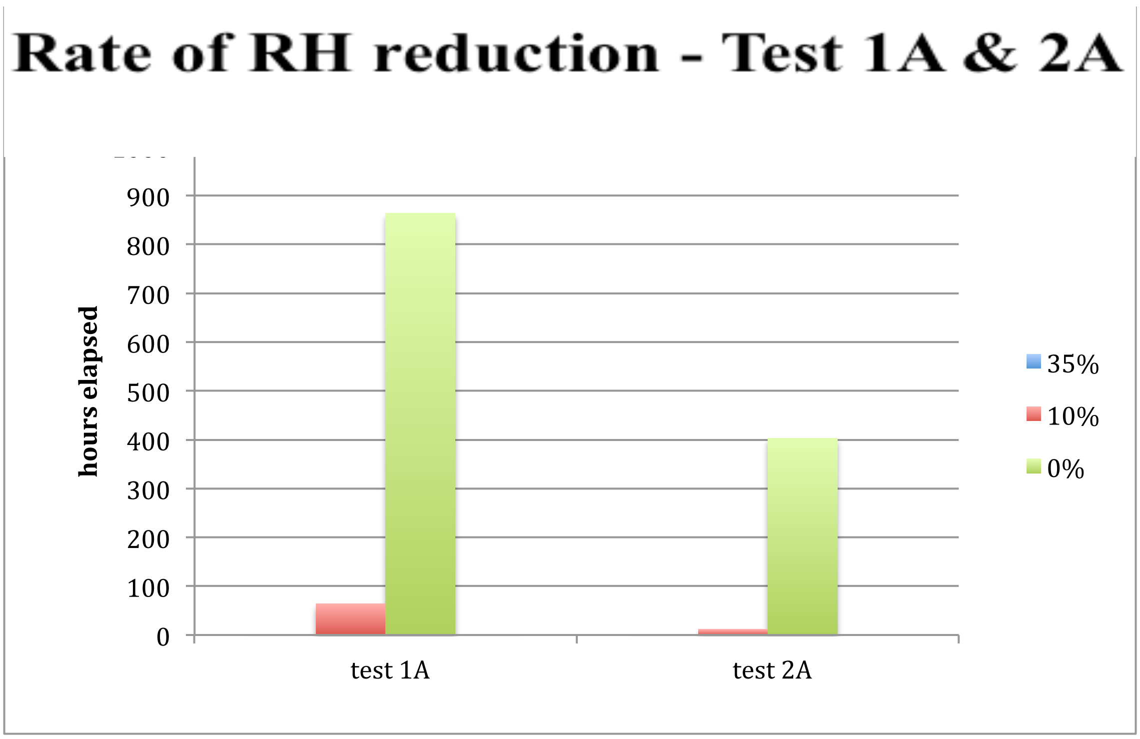

Figure 5 rate of RH reduction to 35%, 10%, and 0%

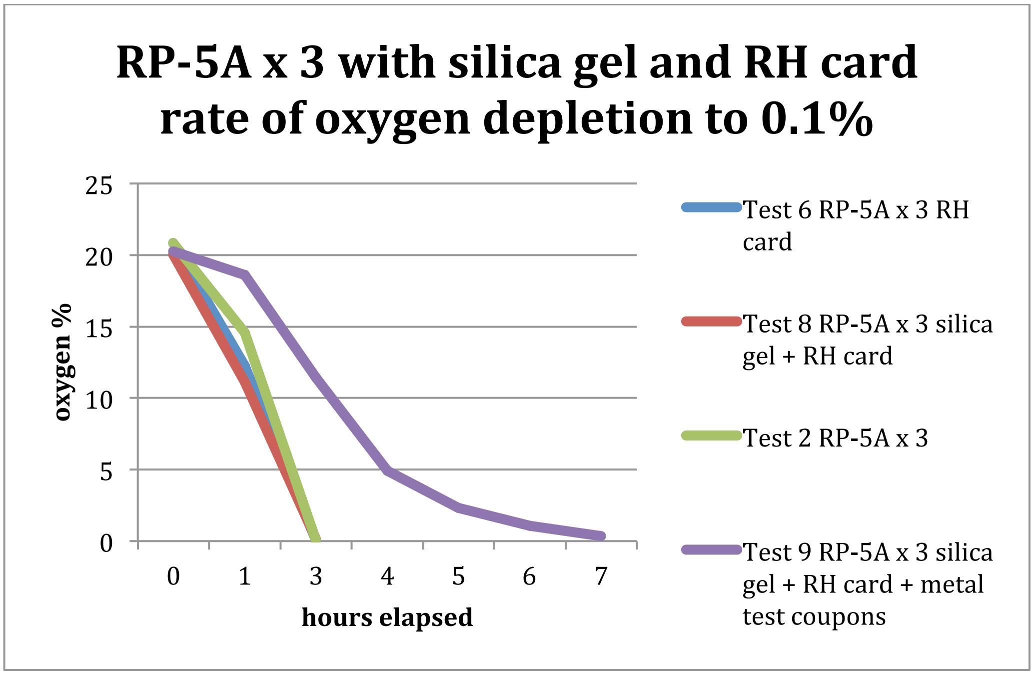

Figure 6 impact of humidity indicator card with and without silica gel on oxygen depletion

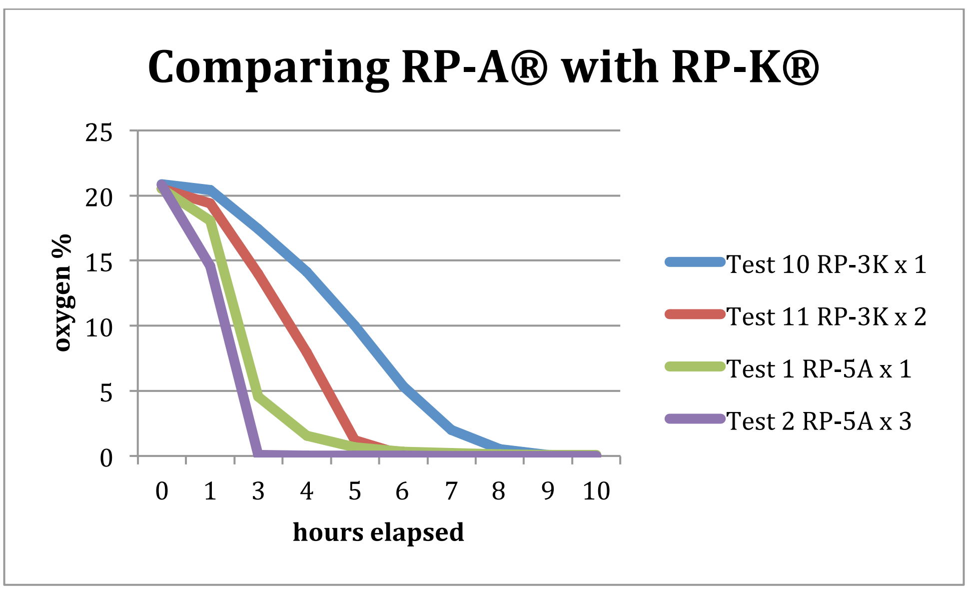

Figure 7 comparing rate of oxygen depletion by RP-A and RP-K

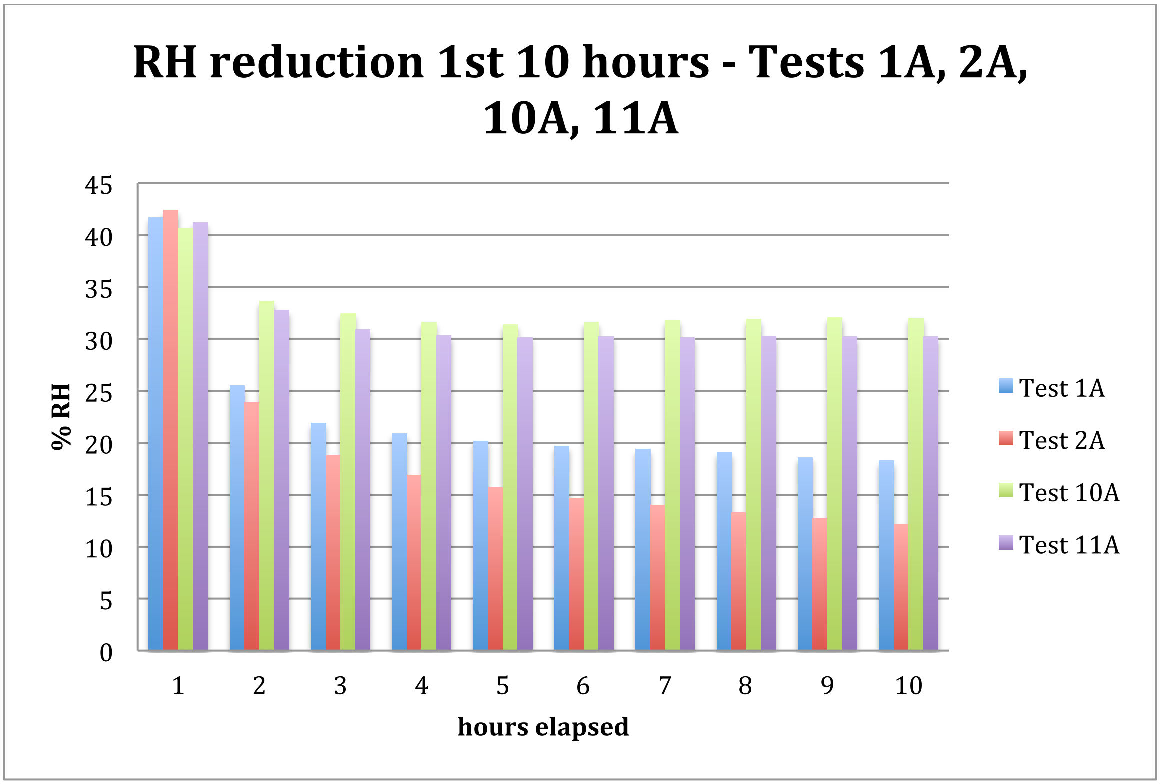

Figure 8 RH reduction first 10 hours - Tests 1A, 2A, 10A, 11A

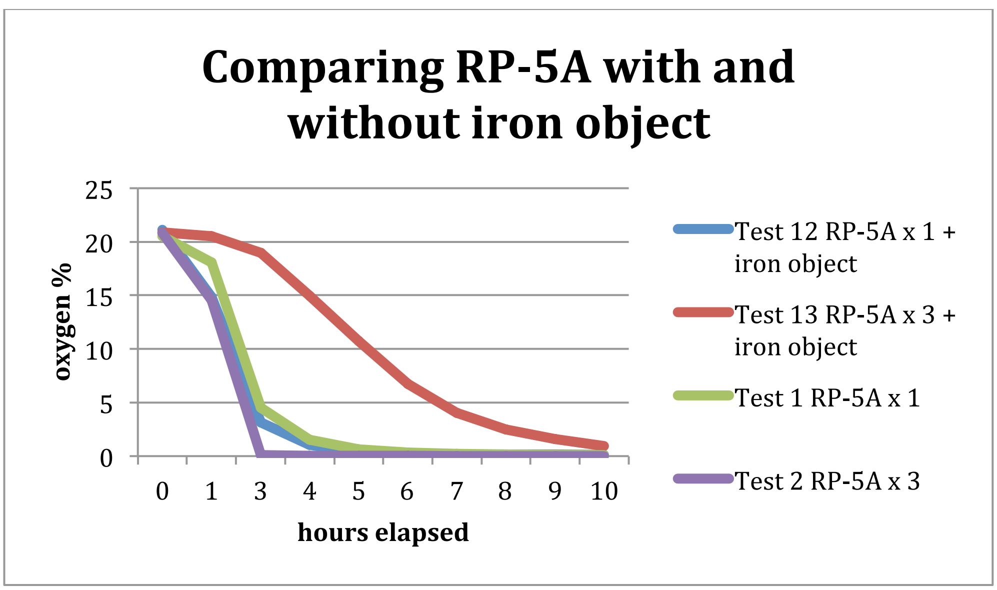

Figure 9 impact of uncleaned iron object on oxygen reduction to < 0.1%

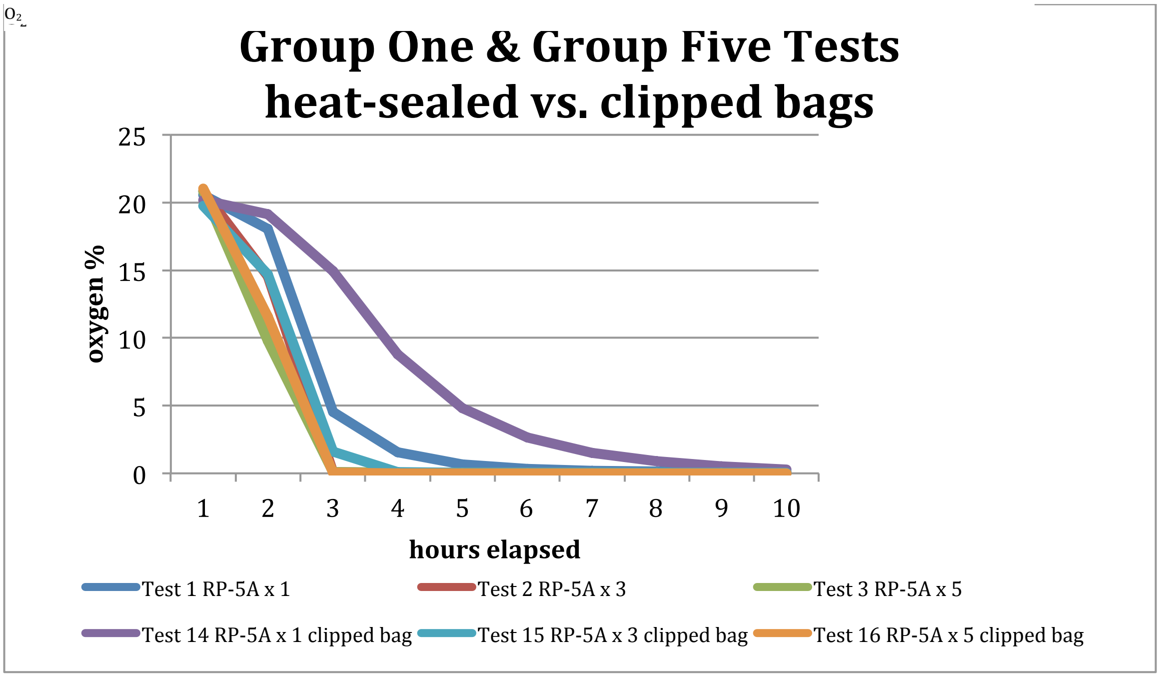

Figure 10 comparing heat-sealed and clipped Escal bags

Discussion

The results from the previous tests that monitored the rate of moisture absorption by RP-A and silica gel prompted further testing (Paterakis & Mariano 2013). Likewise, the results of the tests that monitored the rate of oxygen absorption by RP-K, based on color change of the oxygen eye indicator, are considered to be inconclusive (Paterakis & Mariano 2013). The fact that RP-K was found to suppress the RH from reaching the target of 60% RH for 23 days in the 2013 tests warranted further investigation. Since increasing the ratio of silica gel weight to air volume was found to accelerate the rate of moisture absorption it was considered expedient to test the impact of the ratio of oxygen scavenger weight to air volume on oxygen absorption. The oxygen content was monitored efficiently in the current tests with the MOCON OpTech-O_2 Platinum oxygen meter.

Influence of iron corrosion

A comparison of Test 2 and Test 13 shows that uncleaned iron had a major impact on oxygen absorption: Test 13 with an uncleaned iron object required more than 10 hours to reach < 0.1% O_2 (23° C) (Figure 9) (Table 1). Likewise, iron objects were shown to retard the rate of oxygen absorption in the 2013 tests (Paterakis and Mariano 2013).

Comparing the RPA with the RPK

Although the bag sizes differed between Tests 1 and 2 with RPA and Tests 10 and 11 with RPK, the manufacturer's recommended ratio of sorbent size to air volume of bag was followed in Test 2 and Test 11 (Table 1). With this knowledge, the reduction of oxygen content to <0.1% in three hours and to 0.015% in six hours by the RPA (24° C) compared to six hours to reach <0.1% and eight hours to reach 0.015% by the RPK (24° C) may be taken as an indication of RPA's greater suitability as a stabilizer for freshly excavated iron (Figure 7).

Influence of cobalt-containing humidity indicator cards and cobalt-indicating silica gel

When comparing Test 2, Test 6, and Test 8, there is an indication that the humidity indicator cards, with or without the silica gel, may have increased slightly the rate of oxygen reduction to < 0.1% O_2 (23.5°- 24° C) (Figure 6).

Monitoring oxygen depletion

Moisture from the iron objects (Group 4, Tests 12 and 13) and cobalt chloride from the humidity indicator cards may have interfered with the color change of the RPS oxygen indicator eyes; they were still purple in Test bags 13B and 13C when the MOCON oxygen reading was less than 0.1% (23° - 24.5° C). RPS oxygen indicator eyes are supposed to change from purple to pink when the oxygen concentration level dips below 0.1%.

Conclusions

Preliminary results indicate an increased initial rate of oxygen absorption with increasing ratio of RP-A and RP-K scavenger weight to air volume (Figure 4). Exceeding the recommended number of scavengers, however, was found to retard oxygen absorption from <0.1% to 0% O_2 (Table 1). Freshly excavated and untreated iron significantly retarded the rate of oxygen absorption in the Escal bags indicating that it may be necessary to exceed the number of recommended RP-A scavengers for the anoxic storage of uncleaned iron in Escal bags. It should be kept in mind that chloride in corroded iron can influence the oxygen content in the Escal bag and the absorption of oxygen by the scavengers (Thickett et al. 2011; Matthiesen and Stemann-Petersen 2013).

Whether the cobalt chloride in the humidity indicator cards and silica gel may have influenced the slightly accelerated absorption of oxygen by the RPS scavengers in Tests 6 and 8 is not known. Since the toxic properties of cobalt are well known, substitutions are being made today in the manufacture of humidity indicators and silica gel. However, cobalt containing humidity indicator cards and silica gel are still available on the market and are used in many parts of the world. For this reason further tests comparing cobalt-indicating silica gel with other types of silica gels and desiccants should be carried out to clarify the impact of cobalt on oxygen depletion.

Sealing the Escal bags with the plastic clips provided by the manufacturer has shown that once the 0% RH and 0% O_2 levels are reached, these levels were maintained through 12 months in storage (Figure 2). These clips may be recommended for the temporary storage of artifacts in Escal immediately upon their excavation, as well as before, during, and after conservation treatment. The clips may be opened and closed multiple times allowing for easy access although the manufacturer warns against repeated opening that may eventually weaken the seal. A simple test involves clipping one layer of Escal film and trying to pull it out, a lack of success indicates a good seal and a clip in good condition (Mitsubishi 2016). Once the artifact is deemed ready for more permanent storage, the Escal bag may be heat-sealed. Granted, the quality of the seal, albeit heated or clipped, is crucial for the integrity of the anoxic microenvironment. The RP System may be considered both a temporary means of stabilization prior to conservation treatment as well as a long-term storage solution following conservation intervention (Mathias et al. 2004).

With RPA, concerns regarding the appropriate levels of RH required for the prevention of corrosion and the safe maintenance of iron and bronze artifacts over the long term are diminished since 0% RH and 0% O_2 are reached relatively quickly and maintained in well-sealed Escal over the long term. Comparing RPA with RPK, the RPK depleted oxygen at a slower rate perhaps discouraging its use as a fast method of anoxic stabilization. Taking into account the relative costliness of the sensors, they may be inserted in a small percentage of the total number of storage bags that are prepared at the same time with objects of similar size and condition and stored in the same environment, to serve as oxygen checks for the group while minimizing cost.

With these tests we have contributed further knowledge towards the successful use of the RP System for the protection of archaeological metals. Plans are underway to carry out more tests using oxygen scavengers and silica gels for the storage of archaeological iron and bronze artifacts. The RP-K oxygen scavengers will be tested with preconditioned silica gel and composite objects. RP-A scavengers will be tested with and compared to non-indicating silica gel, orange-indicating silica gel, and cobalt-indicating silica gel. The goal of these tests will be to determine the impact of humidity buffering materials, iron artifacts, and composite objects on the efficiency of the RP-K and RP-A scavengers.

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Materials and Suppliers

Rotronic HL-20 datalogger, Rotronic Instrument Corp., 135 Engineers Road, Suite 150, Hauppauge, NY 11788, www.rotronic-usa.com/

HOBO U10 datalogger, Onset Computer Corporation, 470 MacArthur Blvd., Bourne, MA 02532, http://www.onsetcomp.com/products/data-loggers/u10-003

Humidity Indicator Card, Süd-Chemie Performance Packaging, Colton, CA, available from Hollinger Metal Edge, http://www.hollingermetaledge.com/

RP System: RP-A and RP-K scavengers, Escal, oxygen indicator eyes, and clips available from Mitsubishi Gas Chemical America, Inc., 655 3rd Ave., New York, NY 10017, http://ageless.mgc-a.com/product/rp-system/

MOCON OpTech-O_2 Platinum oxygen meter, MOCON Inc., 7500 Mendelssohn Ave. N., Minneapolis, MN 55428, www.mocon.com

Heat-sealer, FoodSaver V3240 Vacuum Sealer, www.foodsaver.com/index.aspx www.target.com

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