



Decontamination of chilli flakes in a fluidized bed using combined technologies: Infrared, UV and ozone



Ian Watson^{a,*}, Prashant Kamble^a, Callum Shanks^a, Zakir Khan^{a,b}, Nada El Darra^c

^a Systems, Power and Energy Research Division, School of Engineering, University of Glasgow, James Watt (South) Building University Avenue, Glasgow, UK

^b Department of Chemical Engineering, COMSATS, University of Islamabad, Lahore Campus, Lahore, Pakistan

^c Beirut Arab University, Faculty of Health Sciences, Tarik El Jedidah, Beirut, P.O.Box: 115020 Riad El Solh 1107 2809, Lebanon

ARTICLE INFO

Keywords:

Infra-red
IR
Ultraviolet
UV
Ozone
Combination
Treatment
Chilli
Chilli
Flakes
Spices
Decontamination
Inactivation

ABSTRACT

An initial study of the combination of IR, UV and ozone on treating raw and artificially inoculated chilli flakes (CF, *Capsicum annum* L) was assessed using a specially designed fluidised bed system, in a fused quartz tube, using a distributor plate and filtered air, to keep the CF in an air suspension. The untreated samples, as bought, were contaminated with $\sim 1 \times 10^6$ cfu/g, they were subsequently autoclaved, and inoculated with *Escherichia coli* (MG1655). The individual treatments were all effective in reducing the bioburden with log reduction and treatment times ~ 6 logs (cfu/g) in ≤ 20 min for ozone ($300 \text{ mgO}_3\text{hr}^{-1}$); ~ 7 logs (cfu/g) in ≤ 40 min, for UV (4 W); and ~ 7 logs (cfu/g) in ≤ 20 min, for IR (100 W); for stationary air with ozone, and an airflow of 108 Lmin^{-1} with UV and IR treatment. The IR was modulated so that the external tube temperature was $58\text{--}60^\circ\text{C}$. The treatment order was more effective for the IR and UV followed by ozone, than ozone followed by UV and IR (ozone, 10 min, UV and IR 10 min combined), this was due to the higher initial reduction of the UV and IR (0.80 log (cfu/g)) than with ozone first (0.13 log (cfu/g)). Such decontamination systems could be used efficiently when conveying the spices, just prior to aseptic packaging.

1. Introduction

Spice and herb commodities are traded worldwide with a value of US\$4 billion per annum; this is expected to grow to US\$6.5 billion in the near future, according to the International Trade Centre (International Trade Centre UNCTAD/WTO, 2006). The European Union is the second largest spice market in the world, importing 533 thousand tonnes of spices and herbs per year (€1.9 billion in 2014). European imports of spices and herbs from developing countries have grown by 6.1% annually between 2012 and 2016 (CBI, 2016). US consumers are also becoming increasingly interested in herbs and spices (Galvin-King, Haughey, & Elliott, 2018). The global, annual spice and seasoning market is valued at US\$15B (ASTA, 2011a) and is heavily reliant on international shipping which places greater emphasis on shelf-life and handling. With such a global importance of spices, their integrity, quality and safety is of high importance.

Spices are mostly dry products, which are naturally contaminated. The cost of contamination of food products leading to food poisoning is estimated to be \$50B per year (Scharff, 2012). Pathogenic bacteria, such as *Salmonella*, *Bacillus cereus*, *Clostridium perfringens*, and

Escherichia coli, are commonly detected on herbs and spices (Banerjee & Sarkar, 2003; Sagoo et al., 2009).

There is continued interest in developing effective methods of decontamination of pathogens and spoilage organisms on spices to eliminate these risks and to increase the shelf-life for these higher value products (Van Doren, Kleinmeier, Hammack, & Westerman, 2013).

Different treatments have been used to decontaminate spices; however, most of these treatments presented some detrimental effects on the food quality. Thermal treatment of spices and herbs, including dry steam, is the most widely used technology to ensure microbiological safety. However, it can cause significant physical and chemical changes that negatively affect sensory and nutritional quality (Baysal, Molva, & Unluturk, 2013; Polovka & Suhaj, 2010). Chemical treatment using ethylene oxide has also been used to decontaminate spices; however, its use was prohibited by an EU directive in 1991 and has been banned in a number of other countries because it is a carcinogen. Gamma irradiation has since emerged as a viable alternative and its use results in cleaner, better quality herbs and spices compared to those fumigated, yet the technique is still restricted in EU countries (Polovka & Suhaj, 2010) and not favoured by consumers. Subsequently, it is of vital socio-

* Corresponding author.

E-mail address: Ian.Watson@glasgow.ac.uk (I. Watson).

<https://doi.org/10.1016/j.ifset.2019.102248>

Received 12 April 2019; Received in revised form 8 August 2019; Accepted 23 October 2019

Available online 24 October 2019

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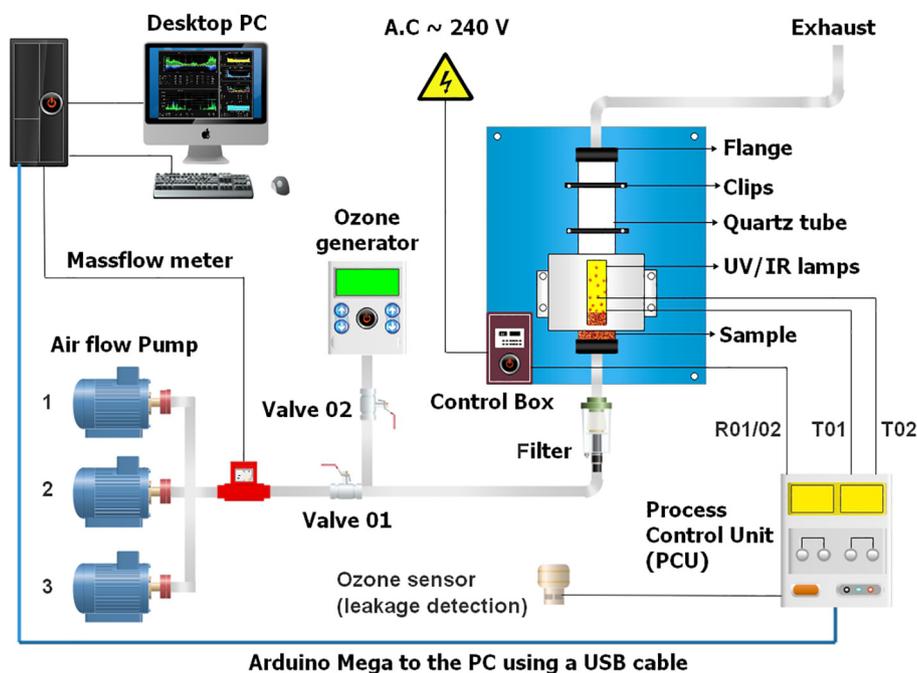


Fig. 1. Experimental set up of the combined ozone, UV, IR decontamination system.

economic importance to investigate alternative methods to effectively decontaminate dried spices, render them safe for consumption and improve market access by maintaining the quality of the product and increasing shelf-life. With the large volume of spices consumed globally, there are obvious market gaps in deploying more favourable technology for spice decontamination and extending their shelf-life; the demonstrated system and subject of this paper, demonstrates a viable and cost-effective option.

A combination of non-thermal methods with antimicrobial treatments can enhance the lethal effects of non-thermal processing, reduce the severity or the exposure time of non-thermal treatment needed to obtain a given level of microbial inactivation and preserve the food's physio-chemical properties without affecting the nutritional value (Raso & Barbosa-Cánovas, 2003).

Ozone is a decontamination treatment that has been used for vegetables and dried foods such as cereal, grains, black pepper and pistachios (Akbas & Ozdemir, 2006; Khadre, Yousef, & Kim, 2001). It has been approved as an antimicrobial agent for foods in the US, owing to its powerful oxidizing effect (Guzel-Seydim, Greene, & Seydim, 2004). Ozone treatment was found to be effective against a wide spectrum of microorganisms, including viruses, gram-negative and gram-positive bacteria, spores and fungi (Manousaridis et al., 2005). Its use showed a decrease in the bacterial load of *Bacillus* spp. and *Micrococcus* counts in dried foods and spices by up to 6 log numbers (Zhao & Cranston, 1995). Ozone at 1 ppm was found to be effective in reducing by 3.5 log numbers the *E.coli* counts in pistachios (Akbas & Ozdemir, 2006). Using ozone gas at 5.3 mg/L for 90 min significantly reduced aerobic plate counts by > 3.2 log in oregano (Torlak, Sert, & Ulca, 2013).

Ultraviolet light is another technique that has shown lethality effects on different microorganisms, such as bacteria, yeasts, molds and viruses. It was also approved as a decontamination treatment for food surfaces (Baysal et al., 2013). The decontamination effect of the UV is mainly due to the ability of the light to penetrate the cell wall, thus blocking DNA transcription and replication, which inhibits the microorganism's ability to grow and proliferate (Azimi, Allen, & Farnood, 2012; Chun, Kim, Lee, Yu, & Song, 2010). A study by Yaun, Sumner, Eifert, and Marcy (2004) showed that the treatment of fresh produce by ultraviolet energy was effective at reducing microbial loads of pathogens on fresh fruits and vegetables. UV is a cheap, easily implementable

technique, which doesn't generate chemical residues and presents a lethal effect against a broad range of microorganism (Baysal et al., 2013).

Infrared (IR) decontamination treatment has been reported for its effectiveness in many spices. This was mainly due to their rapid heating capacity, with significant energy saving, resulting in less heating time and better quality preservation compared to conventional heating methods (Krishnamurthy et al., 2008). IR heating has produced promising results for the surface pasteurization of black pepper seeds (Erdoğan & Ekiz, 2013), as well as for cumin seeds if combined with ultraviolet treatment (Erdoğan & Ekiz, 2011). Moreover, Staack (Staack, Ahn, Borch, & Knorr, 2008) found that IR treatment efficiently decontaminated *B. cereus* spores in paprika powder.

Among spices, Chilli (*Capsicum annum* L.), also called red pepper is the second largest consumed spice throughout the world (Santos, Marín, Sanchis, & Ramos, 2010). Chilli, belonging to the night shade family of Solanacea, is mainly cultivated in developing countries with tropical and/or semi tropical climates and exported worldwide (Santos et al., 2011).

Few, if any, studies have assessed the performance of combined systems to decontaminate spices. Therefore, the purpose of this work is to evaluate a novel, non-thermal combined technology of ozone, UV-C radiation and modulated IR light as a decontamination treatment for chilli flakes (CF) artificially inoculated with *Escherichia coli*. The system incorporates a fluidized bed that suspends and moves the chilli flakes in the region of the IR and UV energy in air. The results obtained in this work may serve as basis in developing a combined decontamination system's approach for spice decontamination and other foods, such as powders, that can be fluidized or suspended in an air flow.

2. Materials and methods

2.1. System description

The system (Fig. 1) comprised a 900-mm long fused quartz tube, mounted vertically, with ID of 33 mm and wall thickness of 3 mm. An air pump (MA100-120, Jecod, UK) provided an unrestricted air flow rate of up to 120 L/min which reduced to approximately 108 L/min in the tube, the flow rate was controlled with a mass flow meter (Red-Y,

Vogtlin, Germany). Attached at the lower end of the tube was a distributor plate which was designed to create an even flow of air over the cross-sectional area of the tube to allow suspension of the chilli flakes. Calculations were done to estimate the size of the holes in the distributor plate using fluidized bed theory. The height of the suspended particles varied over time, with flakes rising and falling when the velocity is above a minimum threshold.

The distributor plate was fitted into a mounting-lid that was secured in place at the bottom of the tube via an O-ring, sealing the tube. The samples were placed on top of this aluminium insert; the lid could be easily removed and reinserted facilitating simple sample insertion and extraction. The samples of CF were added to the tube aseptically and the tube was wiped before and after each insertion. A standard germicidal UV (4 W) and IR lamp (100 W, SK15 Jacketed, Victory Lighting, UK) were placed symmetrically opposite each other approximately, ≤ 25 cm from the base of the tube, and surrounded by a metal cover with a polycarbonate window for safety. The UV and IR lamps were individually controllable (on/off). The UV lamp consumed approximately 15.53 ± 0.02 W ($n = 10$, SEM) after 5 min of turning on which increased to 16.23 ± 0.12 W after an hr. The IR lamp consumed 110.78 ± 0.12 W from 30 s from turning on and remained stable over its period of operation. The UV and IR lamp were each 20 mm from the side of the quartz tube.

A temperature sensor (TEMPer1F, PC Sensor, China) was located on the outside surface of the tube, away from direct IR exposure; this avoided any internally placed thermocouples disrupting the flow of the contaminated samples. During IR treatment the outside surface temperature of the tube was maintained to approximately 58–62 °C, in this first case manually. In a test case without any samples added to the tube, a temperature probe was placed inside the tube to assess the temperature variation across the tube, with and without flow and the internal and external temporal temperatures were compared. An ozone generator (GMB-DOM-024, GMB Ozone purifier, Italy) was connected to the main flow line and was isolated by a valve, its maximum capacity was $300 \text{ mgO}_3\text{hr}^{-1}$, with a power consumption of 25 W. The flow rate from the ozone generator was only 2–3 L/min which was incompatible with the main air flow from the pump (100 L/h), consequently, the ozone treatment was done without any additional airflow, hence the samples were essentially a loosely packed bed during this treatment.

The relative position of the UV and IR lamps to the fluidized chilli flakes was easily changed by moving the tube up or down or by varying the air velocity. The system air velocity was optimized so that the flakes would spend most of their time within the UV and IR irradiated region, and was 105–108 L/min.

Experiments were done to determine the temporal temperature rise inside the tube at different positions with the IR lamp on over time; this was done in the tube centre, and at positions closest and furthest away from the tube with no flow and no CF. Additionally, experiments were done to measure the temperature inside (middle) of the tube and outside the tube, in a fixed place, whilst modulating the IR lamp, here the flow was on and CF were in the tube.

2.2. Calculation of the minimum fluidization velocity

A fluidised bed imposes an upward drag force from flowing gas on particulates that is greater than gravity. With increasing gas velocity, above the minimum fluidisation velocity, the bed enters different stages of fluidisation from minimum, bubbling to fast and beyond which there is conveying. Particles can be split into 4 Geldart groupings according to their fluidisation capacity C (most difficult to fluidise), A (easiest), B (unsmooth) and D (largest particles); (Cocco, Karri, & Knowlton, 2014). The hydrodynamics of the fluidized bed is initiated by evaluation of the minimum fluidization required to fluidize the chilli flakes. The operating conditions were assumed to be room temperature and 1 bar. The minimum fluidization velocity (U_{mf}) is a basic design parameter to

define fluidization conditions in the bed. It is a function of bed particle diameter, density, shape and fluidizing gas transport properties (Sadaka, Ghaly, & Sabbah, 2002). In different fluidized regimes, the superficial gas velocity is of the order of U_{mf} (Basu, 2006). The modified form of Ergun's equation, in the form of Archimedes number (Ar) for the pressure drop across a fixed bed at minimum fluidization conditions, is used to estimate U_{mf} (D. Kunii, 1992) and given by Eq. (1):

$$Ar = 150 \left(\frac{1 - \varepsilon_{mf}}{\varphi_b^2 (\varepsilon_{mf}^3)} \right) Re_{mf} + \left(\frac{1.75}{\varphi_b^2 (\varepsilon_{mf}^3)} \right) Re_{mf}^2 \quad (1)$$

where Ar can be calculated as:

$$Ar = d_b^3 \rho_f \left(\frac{\rho_p - \rho_f}{\mu^2} \right) g \quad (2)$$

The Reynolds number at the minimum fluidization condition is given by

$$Re_{mf} = \frac{d_p \rho_f U_{mf}}{\mu} \quad (3)$$

where d_b and ρ_b are the bed particle diameter (m) and density (kg/m^3), respectively, μ and ρ_f are the viscosity (Pa. s) and density (kg/m^3) of the fluid, air in this case, g is the acceleration due to gravity (m/s^2), ε_{mf} is the bed voidage at the minimum fluidization velocity and φ_b is the bed particle sphericity. Bed voidage and sphericity of bed particles must be known at minimum fluidization to estimate U_{mf} using Eqs. (1), (2) and (3). These basic equations give more reliable predictions of U_{mf} as compared to empirical expressions (D. Kunii, 1992) and thus they are used herein. ε_{mf} is calculated from the following expression (Rhodes, 1998):

$$\varepsilon_{mf} = \frac{\rho_b}{\rho_p} - 1 \quad (4)$$

2.3. Raw materials

CF (*Capsicum annum* L) were purchased from a local supermarket, originating from China and packaged in London, UK. The flakes were stored at room temperature ($20 \text{ }^\circ\text{C} \pm 1$) in a dry place. One-g samples of the CF were suspended in 9 mL of sterile water and vigorously shaken for 20 s. Successive serial dilutions were taken to ascertain the natural bioburden on the flakes, plated onto Luria Broth (LB) agar plates and incubated overnight at 37 °C. This was repeated in duplicate. Colony-forming units (CFU) were counted and the bacterial concentration on the CF determined per gram. Please note that the base contamination levels were high ($> 2 \times 10^6$ CFU/mL) which necessitated inactivation and inoculation with a controlled level of bacteria. The inactivation process, therefore, rendered any nutritive or sensory analysis non-valid and subsequently this was not investigated in this preliminary analysis of the biocidal efficacy of this system.

2.4. Bacterial strain preparation and sample inoculation

An overnight culture of *Escherichia coli* (MG1655) was supplied by the Environmental Engineering Laboratory, School of Engineering, at the University of Glasgow. Different methods of sterilizing the CF were tested before inoculation. The test methods included: autoclaving, soaking in ethanol and microwaving. Two-g samples of the raw CF were subjected to the treatments; after which 1 g was placed in 9 mL of sterile water and serial dilutions were done in distilled water, tests were done in duplicate.

2.5. Pretreatment of the chilli flakes

2.5.1. Autoclaving

The CF were autoclaved in a portable pressure steam sterilizer

(model YX280, Getty, China), 2 cycles were tested at a temperature 121 °C and a treatment time of 2 or 10 min and the visual appearance of the chilli flakes was assessed and compared to untreated samples. One-g was placed into 9 mL of sterile water for subsequent serial dilutions followed by plating and incubation.

2.5.2. Soaking in ethanol (70%)

Two-g of CF were soaked in 8 mL of ethanol and agitated for 30 s, and repeated in duplicate. After agitation the ethanol was decanted and the samples were washed twice with sterile water. A 1 g sample was taken for serial dilution.

2.5.3. Microwave

Five-g samples of CF were placed into two different glass beakers, which were individually subjected to microwave (Model MTG06, Tesco, UK) treatment (1200 W rating) for a period of 1 min. Following exposure, 2 × 1 g samples were separately diluted into 9 mL of sterile water and serial dilutions performed.

The three methods of sterilization were compared to the counts from the raw material and the most efficient decontamination method was chosen as the preferred method in the study before inoculation of a standard sample.

2.6. Inoculation of the samples

Ten-mL of the *E. coli* overnight culture was pipetted onto 20 g of the sterile CF and mixed thoroughly and samples (3x1g) analysed for counts. The samples were dried in an oven at 35 °C over 2 h. Once dried, counts were recovered from the samples and for every 30 min for a further 2 h to identify the natural reduction in bioburden on drying. Once dried, the inoculated CF were exposed to different individual treatments from the system (see Fig. 1): ozone, UV, IR and combinations of the 3 treatments.

The individual treatments were up to one hour for ozone, UV and IR, with samples taken at 0, 2, 5, 10, 20, 40 and 60 min. Different combinations were tested; this included 1) Ozone for 20 min followed by a combination of UV and IR for 20, 40 and 60 min for the inoculated and raw CF; 2) for inoculated samples, a combination of UV and IR for 10 min, followed by ozone for 10 min and 3) Ozone for 10 min, followed by a combination of UV and IR. It should be reiterated that with the ozone treatments the main flow line for air was not activated because of the flow differentials and in this system design it was not possible to combine ozone with the UV or IR. The ozone treatment was then, on a loosely packed, fixed bed of the CF.

3. Results and discussion

3.1. System overview

Fig. 2 shows the temperature profile with respect to time for the middle of the tube and points closest (right) and furthest away from the IR lamp, with the flow off. Interestingly, the temperature rose steadily for each position for about 1 min, with a slightly higher temperature further away from the IR lamp, and then the point nearest the lamp began to rise less steeply. The middle and furthest point were approximately the same temperature after 2 min (72 °C) but the closest point was about 10 °C cooler. This effect is likely due to the tube focussing the IR radiation. It should be noted that with the fluidised bed, the CF are distributed randomly through the tube over time so these small temperature variations are likely to have little difference but the CF may cycle between a hot and cooler region through self-shading.

To reduce the impact of the rising temperature, the IR lamp was modulated. Fig. 3 shows the temporal variation in temperature between the inside centre of the tube and outside of the tube, over a 2-min period with the flow on (108 L/min) and the IR lamp manually

modulated to maintain an external temperature of 60 °C. It is seen that the temperature rises in both cases over a 15-s period and then stabilises to approximately 60 and 37 °C for outside and inside the tube respectively. During experiments with CF the internal temperature was not measured but the external temperature was modulated to between 58 and 60 °C, ensuring that the internal temperature was below any likely threshold for damage to organoleptic properties of the CF.

Temperature measurements inside the tube with CF were about 5 °C higher than without, this was probably due to an increased heat transfer and higher absorption coefficient. With the experiment on contaminated CF, however, the temperature was measured externally to avoid flow interference from the temperature probe and wires, and manually modulated to an external temperature of between ≈ 58–62 °C, indicating (from Fig. 3) that the internal temperature was likely between 35 and 40 °C.

The course CF were found to be nearest to group D for Geldarts group but not obeying the equations, probably due to their irregular shape, however, the height of the CF was controllable with flow rate and they could be raised sufficiently high to sit within the IR and UV irradiated region. Crushed black pepper was also tested for its flow characteristics and behaved according to theory (results not shown).

3.2. Sterilization of the Chilli Flakes (CF) before inoculation

Different methods were tested to sterilize the CF in order to obtain sterile raw flakes before proceeding with the *E. coli* inoculation. The raw CF were found to be heavily contaminated with bacteria ($> 2 \times 10^6$ cfu/g). The raw CF were autoclave treated ($T = 125$ °C, holding time $t = 2$ min & 10 min), soaked in ethanol (70%, $t = 30$ s) and treated with microwave radiation ($t = 1$ min). The microwave treatment was ineffective with no reduction in the bacterial load, the ethanol soaked CF demonstrated a bioburden of about 44×10^4 cfu/g after treatment. However, for the autoclaved samples at 2 and 10 min, no bacterial loads were detected. To minimize the detrimental impact of the autoclave treatment on the quality, with respect to organoleptic characteristics of the CF, the 2 min autoclaving treatment was selected for pretreatment before subsequent *E. coli* inoculation.

3.3. Drying kinetics of CF

The CF samples were inoculated with *E. coli* at a concentration 1.56×10^6 cfu/mL, equivalent to a log count of 7.2 cfu/g. The CF samples were oven dried after inoculation until they were visibly dry. The reduction curve in the surviving *E. coli* population on the CF was monitored during the drying process at different times ($t = 0, 30, 60, 90, 120$ min) in duplicate, and can be seen in Fig. 4 (note the error bars are not observable); there was only a 55% reduction in viability over 2 h, which was sufficient for subsequent experiments. This is similar to the results from a study conducted by (Eze & Agbo, 2011) on the effect of drying on ginger, which showed a reduction of 57.6% of the total viable count for the samples subjected to open-air drying. The inactivation effect of drying on the microorganisms is likely due to reduced water activity (Bourdoux, Li, Rajkovic, Devlieghere, & Uyttendaele, 2016).

3.4. Individual treatment for inoculated CF

Fig. 5 shows the log reduction in the bacterial counts for the individual treatments, a) ozone, b) UV and c) modulated IR, controlled to maintain an external temperature of approximately 60 °C. It is clearly seen that all of the treatments were effective for long exposures, producing complete inactivation of the *E. coli* inoculated on the CF after < 20, < 40 and < 20 min for ozone, UV and IR respectively. It should be noted that in this initial work the exact time of complete inactivation was not found precisely. Ozone was shown to be highly effective against *E. coli* on the CF. This result agrees with the study of

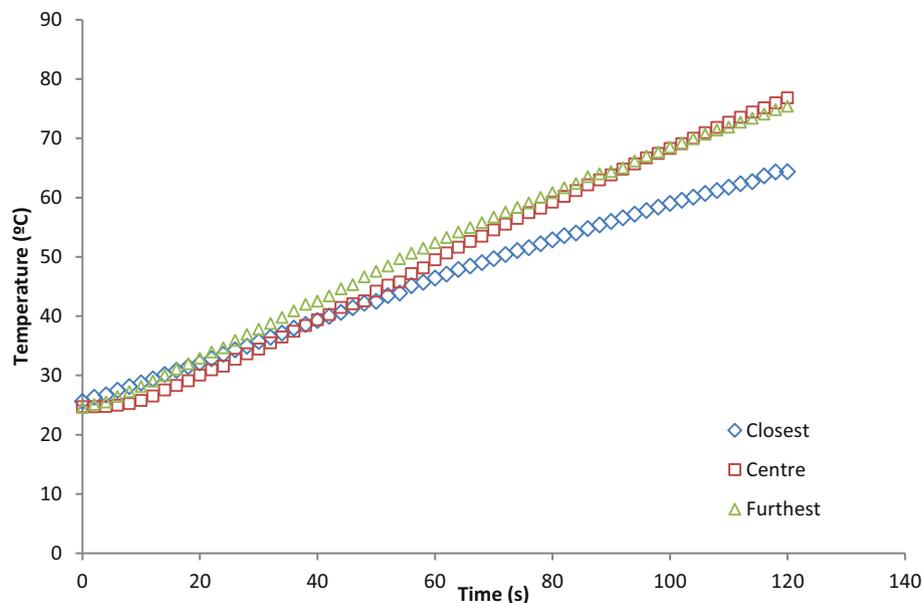


Fig. 2. Internal tube temperatures, furthest (left of tube), tube centre and closest to the IR lamp. (Error bars not shown for clarity. Each point is the average of three readings, the average SEM across all of the data was 0.72, 1.12 and 0.24 °C for the furthest, centre and closest point).

Kazi et al. (Kazi, Parlapani, Boziaris, Vellios, & Lykas, 2018) which showed that ozone is an appropriate disinfection method to be used for the reduction of bacteria on dried aromatic plants which is in agreement with this work. The ozone decontamination effect is due to its ability to destroy a crucial part of the bacterial membrane barrier, consequently leading to the disintegration of the cell wall, and eventually cell death (Karaca & Velioglu, 2014; Mahapatra, Muthukumarappan, & Julson, 2005). *E. coli* was found to be susceptible to low concentrations of ozone, due to its thin peptidoglycan lamella that is covered by an outer membrane made of polysaccharides and lipoproteins (Zuma, Lin, & Jonnalagadda, 2009).

Similarly as with ozone, IR was shown to be efficient in decontaminating the CF. This could be due to the thermal effect of the IR, that will raise the surface temperature of the spices, leading to inactivation of the microorganisms, as shown by the studies of Huang (2004) and Erdoğan and Ekiz (2011). A study examining the effect of IR on the microbial decontamination of paprika powder showed a 1–2 log reductions of microbial flora (Staack et al., 2008).

Fig. 5 shows that UV is also efficient as a single treatment. However,

it requires approximately twice as long as ozone and IR (40 compared to 20 min) for the concentration and energy densities selected for the treatments. The decontamination effect of UV is mainly due to the photochemical oxidation and pyrimidine dimer formation in DNA strands (Miller, Jeffrey, Mitchell, & Elasri, 1999). This alters normal operation of transcription and cell replication. Thus, consequently, resulting in cellular function loss and cellular death (Allende, McEvoy, Luo, Artes, & Wang, 2006). UV appears less efficient than the other two treatments but it should be noted that the IR/UV power consumption was approximately 7.0, and the rated power ratio of the lamps is 25 (IR/UV). This result with the UV irradiation is in agreement with the study from Cheon, Shin, Park, Chung, and Kang (2015), studying the effect of UV-C irradiation on the inactivation of foodborne pathogens in powdered red pepper, they showed that the number of surviving cells exposed to UV-C irradiation treatment was only slightly reduced. Another study conducted on inoculated fresh-cut lettuce with *E. coli* showed a reduction in *E. coli* numbers with increasing UV-C intensity (Kim et al., 2013). However, decontamination processing of spices with only UV-C irradiation had not shown a notable effect (Cheon et al.,

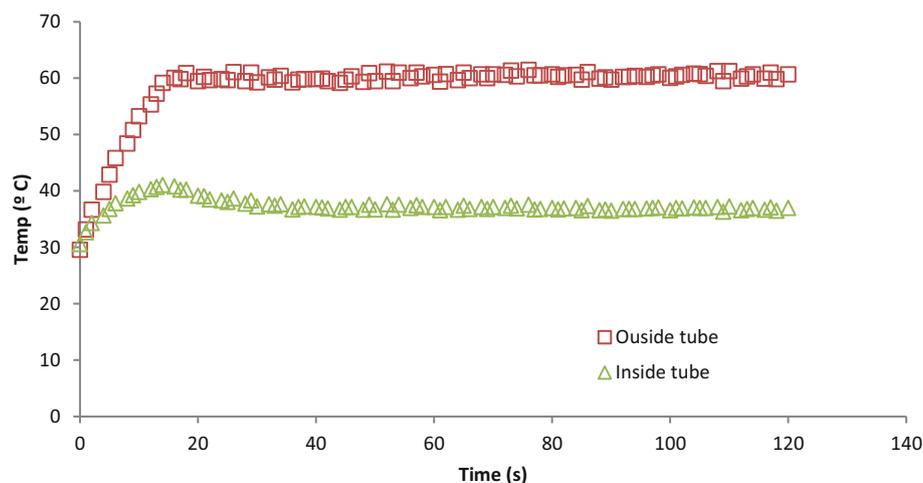


Fig. 3. Centre temperature inside the tube (bottom) and external temperature (top) as a function of time, with IR modulation to maintain 58–62 °C temperature outside the tube, air flow rate of 108 L/min (temperature variation, SD $\leq \pm 4$ °C, $n = 3$).

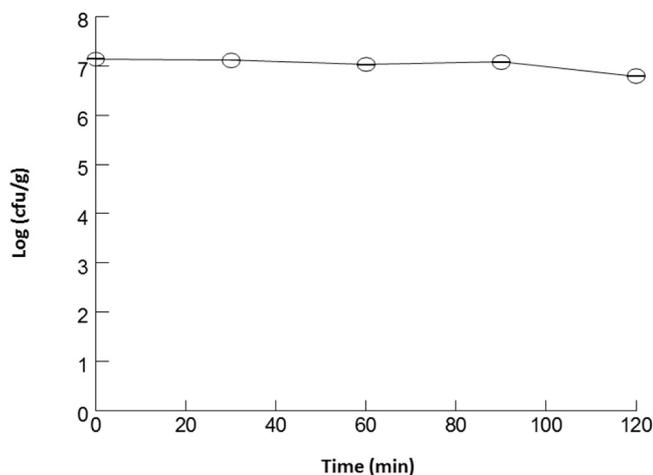


Fig. 4. *E. coli* population kinetics, recovered log (cfu/g) from the inoculated CF over the drying time ($t = 0, 30, 60, 90$ & 120 min). Each point is the average of 3 readings error bars not observable.

2015). To the authors knowledge, no study has investigated the decontamination of spices using combined treatments of UV, IR and ozone whilst the spices are in suspension or in a fluidised bed.

3.5. Combination treatments

Microorganisms contaminating spices reside on the surface with the subsurface parts generally free from contaminants (Erdoğdu & Ekiz, 2011). There have been numerous studies of combined treatments; the lethal effect of combining ozone and UV-C to treat *Bacillus subtilis* spores in a water context was studied by Jung, Oh, & Kang, 2008. It was found that this combined treatment (Ozone concentration = $3.36 \text{ mg l}^{-1} >^1 \text{ min}$ and UV energy density of $14 \text{ mJ cm}^{-2 >^2}$) enhanced the inactivation of the target microorganism by 0.8-log reduction, which corresponded to 33% of the total log reduction (Jung et al., 2008). Another study conducted by Hadjok, Mittal, and Warriner (2008) has shown that an optimized UV-H₂O₂ treatment (1–5% v/v H₂O₂ at 50 °C, $37 \text{ mJ cm}^{-2 >^2}$ UV dose) can enhance the inactivation of bacteria on and within the fresh produce by 2.84 ± 0.34 log CFU, which was significantly higher than individual H₂O₂ or UV treatments. A study by Maktabi, Watson, and Parton (2011), showed a synergistic lethal effect of UV, laser and microwave radiation, with a reduction of 0.38 and 1.06 log in viable counts for the sum of the treatments alone and in combination respectively. Additionally, there were benefits to combining Nd:YAG laser radiation and UV light for decontaminating *Bacillus cereus* spores on agar surfaces, stainless-steel and water (Armstrong, Watson, & Stewart-Tull, 2006), where the effects were at least additive. Such an additive process or synergistic effect is beneficial if increased killing is observed without further degradation of the material substrate.

Fig. 6 shows the effect of combining treatments for a) inoculated

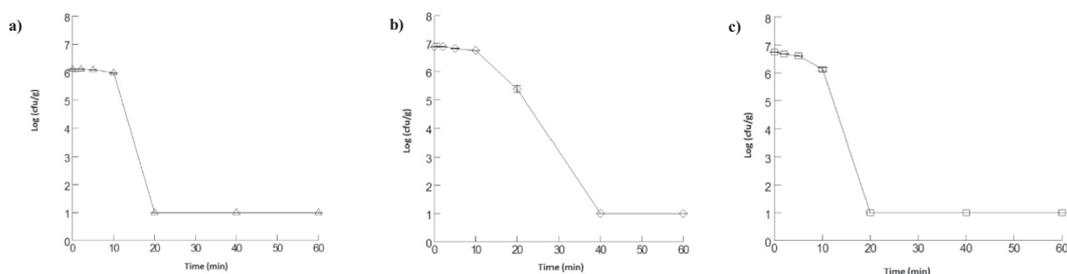


Fig. 5. *E. coli* population kinetics, log (cfu/g) recovered from inoculated CF after individual treatments by (a) ozone, (b) UV and (c) IR for different durations ($t = 0, 2, 5, 10, 20, 40$ & 60 min). Error bars, SD, $n = 2$.

and also b) raw CF. The slight difference in the starting bioburden can be seen in the figure between the artificial inoculation and the samples. In this case, the treatment order was 20 min for ozone, followed by a further 60 min for UV and IR combined. In both cases, it is clear that the ozone had a dominant effect and inactivated all of the bacterial load within this initial treatment window, future work will investigate the impact of reduced ozone treatment times which has a positive impact for potential industrial applications for this process.

To investigate the effect of the treatment order, a reduced exposure time of 10 min was used for the ozone and UV and IR combined, but reversing the treatment order. The results can be seen in Fig. 7, where in a) the UV and IR combined and modulated treatment (10 min) was given first followed by the ozone (10 min), and in b) the ozone was given first, followed by the UV and IR with the same treatment times and conditions. Interestingly, the log reduction was greater for the initial UV and IR combination (a) than for ozone alone in (b). The impact of the second treatment was sufficient to cause complete inactivation of the *E. coli* over the total treatment time for both treatment orders. Clearly, the UV and IR individual treatments alone produced a lower rate of killing than the ozone treatment, as can be seen in Fig. 5. Consequently, it is seen the combination of the UV and IR had a synergistic effect over their individual treatments, and their combined effect was greater than ozone alone. Our results are in accordance with a study (Ha & Kang, 2013), which showed that a combination of IR and UV exhibited synergistic bacteriocidal effects on powdered red pepper inoculated with *E. coli* O157:H7. Another study on cumin, showed that combined IR and UV-C treatments reduced total mesophilic aerobic bacteria to an acceptable level (Erdoğdu & Ekiz, 2011). This increased effect through the combination treatments could be due to the damage of the cell envelope related to the synergistic lethal effect of the treatments.

The combination of IR with UV irradiation was found to be suitable for the surface decontamination of CF. IR-UV had a synergistic effect over their individual treatments, with a greater bacteriocidal effect compared to the ozone alone. These results suggest that this new hurdle technique and delivery system can be introduced as an alternative technique to previous methods, such as ethylene oxide fumigation or steam. Further studies exploring the quality impact of this technology on the CF will be conducted and greater information sought on the impacts of combining the treatment.

4. Conclusion

A novel method of decontaminating chilli flakes has been demonstrated using a combination of ozone, UV and modulated IR light, whilst maintaining the chilli flakes in suspension using airflow. The position of the flakes relative to the lamps could be controlled by changing the airflow (L/min) but of course the flow had to be above a critical value (~ 100 L/min in the present case) to ensure that the CF samples were moved within the UV and IR treatment zones. The behaviour of CF in the flow were not accurately predicted by Geldart's theory but crushed black pepper with its more uniform shape behaved

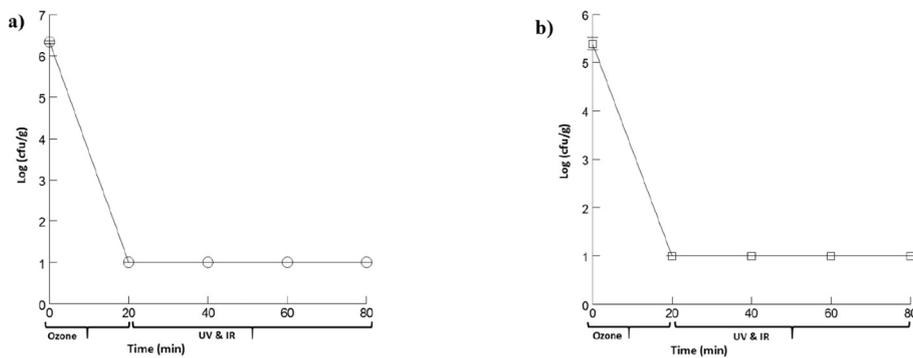


Fig. 6. *E. coli* population kinetics of a) inoculated CF and b) raw CF, showing log (cfu/g), treated by ozone for 20 min, followed by a combined treatment (UV & IR) for 20, 40, 60 and minutes. Error bars, SD, n = 2.

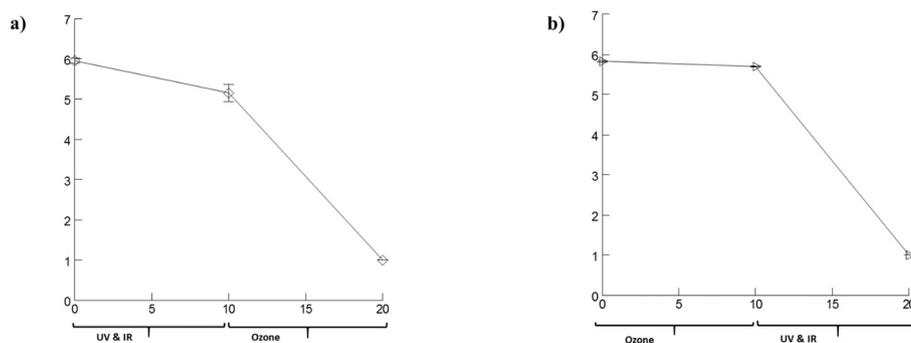


Fig. 7. *E. coli* population kinetics showing log (cfu/g) reduction of inoculated CF after a) UV and IR for 10 min followed by ozone for 10 min and b) Ozone for 10 min, followed by UV and modulated IR for 10 min. Error bars, SD, n = 2.

well. Ozone treatment was done separately to the UV and IR exposures in the current design, with the flakes in a loosely packed bed, as the ozone flow rate was much lower than the air flow rate for suspending the particles. Ozone and IR treatment reduced completely the artificial bioburden ($\sim 1 \times 10^6$ cfu/mL) in < 20 min, whereas for UV the treatment was < 40 min. Combined treatments indicated a slightly better treatment order of UV and IR followed by ozone, than ozone followed by UV and IR, also indicating an improved performance of combining UV and IR than their individual treatments alone. Such systems can be easily retrofitted into food processing lines to ensure that the product is safely decontaminated prior to packaging.

Declaration of competing interest

I can confirm that there is no conflict of interest in this publication with the authors.

Acknowledgements

This work was in part supported by EPSRC (EP/M01343X/1), UK and the Intramural grant, Beirut Arab University, Lebanon.

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